United States Environmental Protection Agency
Air

Office of Air Quality Planning and Standards
Research Triangle Park, NC 27711

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Quality Assurance Guidance Document

Method Compendium

$PM_{2.5}$ Mass Weighing Laboratory Standard Operating Procedures for the Performance Evaluation Program

Foreword

The intent of this document is to describe and provide detailed standard operating procedures for the laboratory activities of the $PM_{2.5}$ Federal Reference Method Performance Evaluation Program (PEP).

The document was developed with the assistance of various workgroups that will be responsible for implementing or overseeing the laboratory aspects of the PEP, as well as State and local organizations that have a vested interest in the quality of the routine ambient air monitoring data. The personnel involved in these workgroups are listed in the acknowledgments.

This document is available in hardcopy format and as a PDF file on the Internet on the Ambient Monitoring Technology Information Center (AMTIC) Bulletin Board under the $PM_{2.5}$ QA area (http://www.epa.gov/ttn/amtic/pmqa.html). The PDF document can be read and printed using Adobe Acrobat Reader software, which is freeware that is available from many Internet sites, including the U.S. EPA web site. The Internet version is write-protected. Hardcopy versions are available by writing or calling:

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Contents

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Acronyms and Abbreviations

AIRS	Aerometric Information Retrieval System
APTI	Air Pollution Training Institute
CFR	Code of Federal Regulations
CMD	Contracts Management Division
CO	Contracting Officer
COC	chain of custody
CS	Contracting Specialist
DAS	data acquisition system
DQA	data quality assessment
DQOs	data quality objectives
EDO	environmental data operation
EMAD	Emissions, Monitoring, and Analysis Division
EPA	Environmental Protection Agency
ESAT	Environmental Services Assistance Team
FEM	Federal Equivalent Method
FRM	Federal Reference Method
GLP	good laboratory practice
LA	laboratory analyst (ESAT contractor)
LAN	local area network
MQAG	Monitoring and Quality Assurance Group
MQOs	measurement quality objectives
NAAQS	National Ambient Air Quality Standards
NAMS	national air monitoring station
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
OAQPS	Office of Air Quality Planning and Standards
OAM	Office of Acquisition Management
OARM	Office of Administration and Resources Management
ORD	Office of Research and Development
PC	personal computer
PE	performance evaluation
PEP	Performance Evaluation Program
$PM_{2.5}$	particulate matter $\leq$ 2.5 microns
PO	Project Officer (Headquarters)
PTFE	polytetrafluoroethylene
QA/QC	quality assurance/quality control
QA	quality assurance
QAPP	quality assurance project plan
QMP	quality management plan
RPO	Regional Project Officer
SLAMS	state and local monitoring stations
SOP	standard operating procedure
SOW	statement or scope of work
STAG	State and Tribal Air Grants
TSA	technical systems audit
WAM	Work Assignment Manager

Tables

Figures

1.0 INTRODUCTION

The purpose of this section is to provide the ESAT laboratory analyst (LA) with background information on the $PM_{2.5}$ Program, and the Federal Reference Method Performance Evaluation Program (PEP) as an introduction to standard operating procedures (SOPs) for laboratory personnel involved in the PEP.

$PM_{2.5}$ Program

In general, the measurement goal of the $PM_{2.5}$ Ambient Air Quality Monitoring Program is to estimate the concentration, in units of micrograms per cubic meter ($\mu g/m^3$), of particulates of aerodynamic diameters less than or equal to 2.5 micrometers (μm) that have been collected on a 46.2mm polytetrafluoroethylene (PTFE) filter in a FRM sampler. In order to understand the size of 2.5 μm, a human hair is approximately 50 μm in diameter. One major objective for the collection of this data is to compare the $PM_{2.5}$ concentrations to the annual (15.0 $\mu g/m^3$ annual arithmetic mean concentration) and 24-hour (65 $\mu g/m^3$ 24-hour average concentration) National Ambient Air Quality Standard (NAAQS). A description of the NAAQS and its calculation can be found in the July 18, 1997 Federal Register Notice. In addition, Appendix L of 40 CFR part 50 also provides the following summary of the measurement principle:

> " An electrically powered air sampler draws ambient air at a constant volumetric flow rate into a specially shaped inlet and through an inertial particle size separator (impactor) where the suspended particulate matter in the $PM_{2.5}$ size range is separated for collection on a polytetrafluoroethylene (PTFE) filter over the specified sampling period. The air sampler and other aspects of this reference method are specified either explicitly in this appendix or generally with reference to other applicable regulations or quality assurance guidance.
>
> Each filter is weighed (after moisture and temperature equilibration) before and after sample collection to determine the net weight (mass) gain due to collected $PM_{2.5}$. The total volume of air sampled is determined by the sampler from the measured flow rate at actual ambient temperature and pressure and the sampling time. The mass concentration of $PM_{2.5}$ in the ambient air is computed as the total mass of collected particles in the $PM_{2.5}$ size range divided by the actual volume of air sampled, and is expressed in micrograms per actual cubic meter of air ($\mu g/m^3$)."

The Federal Reference Method (FRM)Performance Evaluation Program (PEP)

Since the data for the State and Local Air Monitoring Stations and National Air Monitoring Stations (SLAMS/NAMS) network are used for NAAQS comparisons, the quality of this data are very important. Therefore, a quality system has been developed to control and evaluate the quality of data in order to make NAAQS determinations within an acceptable level of confidence. During the development of the $PM_{2.5}$ NAAQS, the EPA used the data quality objective process to determine the allowable measurement system imprecision and bias that would not significantly

effect a decision makers ability to compare pollutant concentrations to the NAAQS. The precision requirement (10%CV) and bias requirement ($\pm$10%) are based on total measurement uncertainty, which incorporates errors coming from all phases (field sampling, handling, analysis etc.) of the measurement process. The collocated samples provide adequate estimates of precision. The FRM Performance Evaluation, if properly implemented, can provide the bias estimate.

The PEP is a quality assurance activity which will be used to evaluate measurement system bias of the $PM_{2.5}$ monitoring network. The pertinent regulations for this performance evaluation are found in 40 CFR Part 58, Appendix A, section 3.5.3. The strategy is to collocate a portable FRM $PM_{2.5}$ air sampling instrument within 1 to 4 meters of a routine NAMS/SLAMS $PM_{2.5}$ air monitoring instrument, operate both monitors, and then compare the results.

The implementation of the FRM Performance Evaluation is a State/local responsibility. However, due to a number of comments made during the review period for the December 13, 1997 $PM_{2.5}$ NAAQS Proposal, the Agency assessed the FRM PEP and consequently made the following revisions:

- modified the system to include an independent FRM Performance Evaluation;
- reduced the burden of this program by changing the audit frequency from all sites to 25% of the $PM_{2.5}$ sites;
- reduced the audit frequency from six times a year to four times a year; and
- made allowances to shift the implementation burden from the State and local agencies to the federal government.

A performance evaluation is defined as a type of audit in which the quantitative data generated in a measurement system are obtained independently and compared with routinely obtained data to evaluate the proficiency of the an analyst or laboratory. In the case of the PEP, the goal is to evaluate total measurement system bias, which includes measurement uncertainties from the field and the laboratory activities. Independent assessment (Figure 0.1) was defined by the $PM_{2.5}$ QA Workgroup in order to ensure that an appropriate level of independence is maintained during State and local implementation of the PEP.

The implementation goal of the $PM_{2.5}$ program is to establish a national monitoring network by December 31, 1999. These sites includes those using FRM/FEM samplers, sites employing continuous analyzers, chemical speciation sites, visibility measurement sites, and special purpose monitoring sites. Each year 25% of the SLAMS/NAMS monitors will be identified for performance evaluations at a frequency of 4 times per year.

During the months of August through October, 1997 the EPA discussed the possibility of Federal Implementation with the EPA Regions, SAMWG and various State and local organizations (NESCAUM, MARAMA, WESTAR, individual organizations). The majority of the responses from these organization were towards federal implementation.

Independent Assessment - an assessment performed by a qualified individual, group, or organization that is not part of the organization directly performing and accountable for the work being assessed. This auditing organization must not be involved with the generation of the routine ambient air monitoring data. An organization can conduct the FRM PE if it can meet the above definition and has a management structure that, at a minimum, will allow for the separation of its routine sampling personnel from its auditing personnel by two levels of management, as illustrated below. In addition, the pre- and postsample weighing of audit filters must be performed by a separate laboratory facility using separate laboratory equipment. Field and laboratory personnel would be required to meet the FRM PE field and laboratory training and certification requirements.

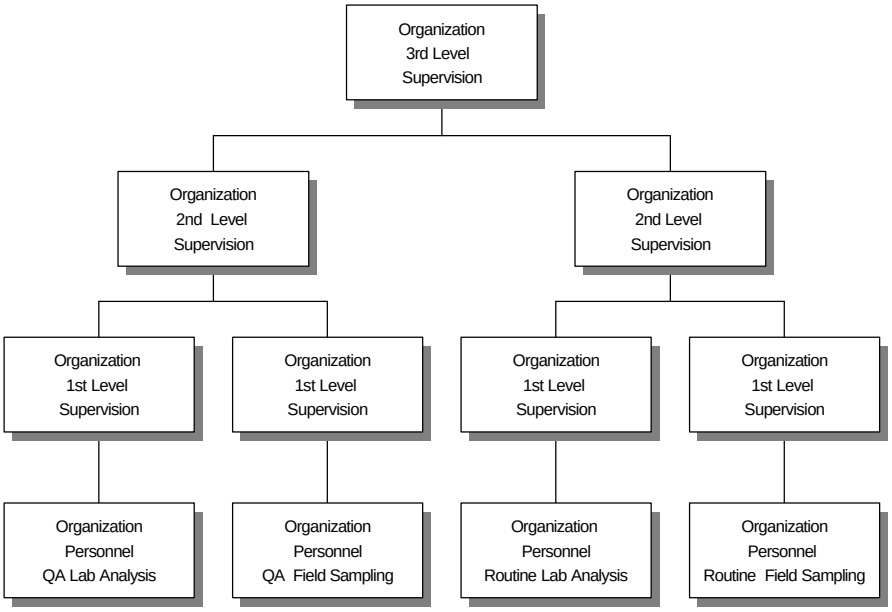

Figure 1

Figure 1.1. Definition of independent assessment

EPA investigated potential contracting mechanisms to assist in the implementation of this activity and will use the Environmental Services Assistance Team (ESAT) contract currently in place in each Region to provide the necessary field and laboratory activities. Each EPA Region will

implement the field component of this activity, while Regions 4 and 10 will also operate the laboratory component.

1.2.3 PEP Activities

The FRM PEP can be segregated into a laboratory component and a field component, followed by data validation and analysis, and then reporting. The following information provides a brief description of these activities. Figure 1.2 provides a basic description of the PEP in five steps:

1. EPA will send filters to Region 4 and 10 laboratories, where they will be checked, equilibrated, labeled, weighed, and prepared for the field.
2. Regions 4 and 10 will ship the filters and accompanying chain of custody (COC) to the Regional field offices.
3. The field scientist will take the filters, field data sheet, and COC to the field and operate the portable samplers.
4. The field scientist will return the filter, data diskettes, field data sheet and COC to the appropriate laboratory (as well as keep a set of the data and records).
5. Region 4 and 10 laboratories will equilibrate and weigh filters, validate data, and upload information to the Aerometric Information Retrieval system (AIRS).

Field Activities:

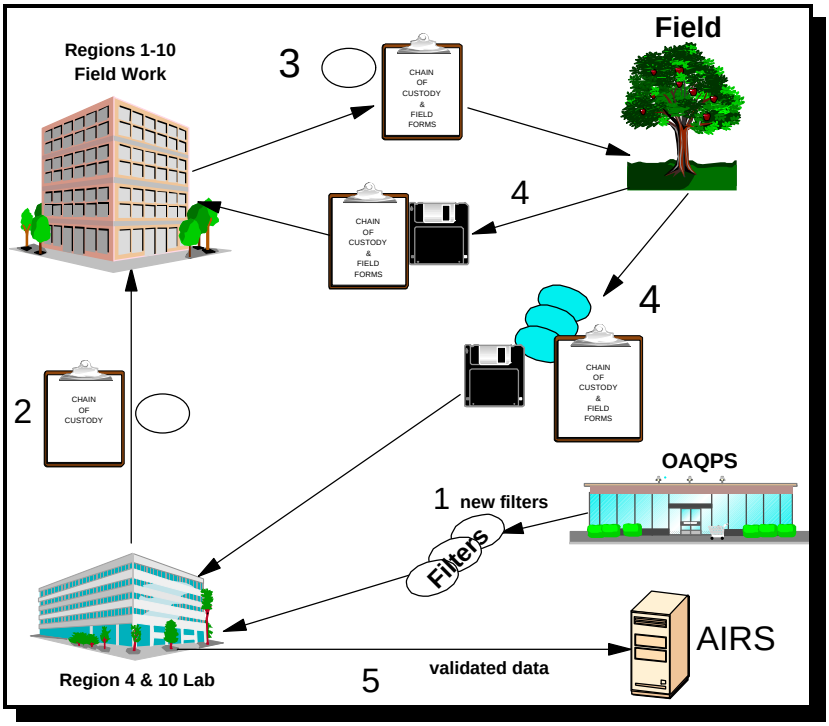

Figure 1.2 Performance Evaluation Program implementation summary

The FRM portable audit samplers will be used in a collocated manner to perform the evaluations. These samplers have been approved by EPA as a Federal Reference Method and are designed to be durable, rugged, and capable of frequent transport. These samplers are constructed in modules with each module weighing no more than 40 pounds. The total weight of the sampler itself must not weigh more than 120 pounds. While these samplers have been specifically designed to perform these evaluations, precautions must be taken

to ensure the quality of the data. Specific detailed instructions will be found in the PEP Quality Assurance Project Plan (QAPP) and SOPs. A brief summary of the field activities follows:

1. One fully trained field scientist will transport a portable $PM_{2.5}$ FRM PE sampling device to an established $PM_{2.5}$ site, which shall be located at any of the SLAMS/NAMS sites within each EPA Region.
2. The field scientist will assemble, collocate the sampler, perform a calibration verification following the SOPs, install a filter and operate the sampler at the required 24-hour sampling schedule (midnight to midnight).
3. If scheduling allows, the field scientist will leave this location to set up an additional 24-hour PE at another SLAM/NAMS sampling location. If the schedule does not allow for another setup, the field scientist may perform additional activities at the site. The field scientist may also perform any required maintenance or repair of the portable $PM_{2.5}$ sampling device followed by a verification to ensure the previous calibration remains valid..
4. The field scientist will return to each site after the 24-hour sampling time, download the stored electronic monitoring data, remove and properly store the filter for transport, and disassemble the instrument.
5. The field scientist will properly package the filter, following the SOPs, for transport to the pre-determined laboratory.

Laboratory Activities:

The FRM PE also requires extensive laboratory activities, including filter handling, equilibration, weighing, data entry/management and archival. Regions 4 and 10 will develop the laboratories for this program. Specific detailed instructions will be found in the PEP QAPP and this SOP. In addition to the good laboratory practices that must be followed, the following activities must also be observed:

- adherence to the vendor's operations manual for the proper operation of the weighing devices; including proper set-up, calibration, and operation of the microbalances;
- adherence to the Standard Operating Procedures (SOPs) for the program;
- adherence to the standards, principles, and practices outlined in the PEP QAPP;
- completion of the required certification training program; and
- giving special attention to any activity involving filter handling (pre-sampling equilibration, weighing, postsampling equilibration, transport, etc.). This area contains the greatest potential for measurement uncertainty, and care must be given to the proper handling of the 46.2-mm Teflon® filter used in the PE.

Presampling weighing:

1. Filters will be received from EPA and examined for integrity based upon EPA approved SOPs.
2. Filters will be enumerated for data entry.
3. Filters will be equilibrated and weighed according to SOPs.
4. Filters will be prepared for field activities or stored according to SOPs.
5. The Laboratory will develop and maintain the shipping/receiving of supplies and consumables, including containers, ice substitutes maximum/minimum thermometers, and COC requirements and documentation.

Postsampling weighing:

1. Filters will be received in the laboratory, checked for integrity (damage-temperature), and logged in.
2. Filters will be archived (cold storage) until ready for weighing.
3. Filters will be brought into the weighing facility and equilibrated for at least 24 hours (per SOPs).
4. Filters will be weighed according to SOPs and data entered.
5. Field data will be entered into the data entry system to calculate a concentration.
6. Filters will be stored in archive for 1 year at ~4 ^{o}C and 2 years at ambient temperature.
7. Required data will be transferred to the AIRS database.

1.3 Purpose of this Document

These FRM PEP Laboratory SOPs provide the detailed procedures to be followed when performing the following laboratory activities, which follow this section as the Lab SOP Section Titles:

- 2.0 General Lab Preparation
- 3.0 Equipment Inventory/Maintenance
- 4.0 Communications (Regions/ State and locals)
- 5.0 Filter Handling
- 6.0 Filter Conditioning
- 7.0 Calibration
- 8.0 Filter Weighing
- 9.0 Filter Shipping
- 10.0 Filter Chain of Custody
- 11.0 Data Entry/Transfer
- 12.0 QA/QC
- 13.0 Storage/Archive

All methods are to be followed completely. Any deviation must be reported in writing and submitted to the ESAT Work Assignment Manager (WAM). Method improvements are encouraged. If any deviations or modifications offer a more efficient method/technique or serve to maintain/improve data quality, these proposed changes shall be made in writing to the ESAT WAM.

Each section will be written as a stand alone procedure that will assist in training and certification activities and can be removed from the document and made readily available at the station where the activity takes place. The SOPs follow the format of the guidance entitled *Guidance for the Preparation of Standard Operating Procedures (SOPs) EPA QA/G-6* for technical SOPs. The QA/G6 requirements include the following topics as Titles:

A. Scope and Applicability
B. Summary of Method
C. Definitions (acronyms, abbreviations and specialized forms used in the SOPs)
D. Health & Safety Warnings
E. Cautions
F. Interferences
G. Personnel Qualifications
H. Apparatus and Materials
I. Instrument or Method Calibration
J. Sample Collection
K. Handling and Preservation
L. Sample Preparation and Analysis
M. Troubleshooting
N. Data Acquisition, Calculations and Data Reduction
O. Computer Hardware and Software
P. Data Management and Records Management.

Each method will address only those topics that are relevant for that method. The methods will be numbered as follows:

PEPL-X.YY

Where:

PEPL	indicates the **P**erformance **E**valuation **P**rogram **L**aboratory SOPs,
X	indicates the section where the method is found (based on the table of contents), and
YY	indicates the method number.

1.4 Prerequisites

1.4.1 Training and Certification

All laboratory personnel funded by the (Office Of Air Quality and Planning and Standards (OAQPS) PEP work assignment (WA) must be trained and certified to perform the activities. Training and recommendation for certification can be provided by the Regional WAM or OAQPS.

1.4.2 Background Reading

Prior to implementing laboratory activities, laboratory personnel are expected to be familiar with the documents listed in Table 1-1. The knowledge level is rated from 1, being very knowledgeable to 5, having a basic understanding.

Table 1-1. Required Reading for the Performance Evaluation Program

Document	Knowledge
FRM Performance Evaluation Program Laboratory SOPs	1
FRM Performance Evaluation Program QA Project Plan	2
FRM Performance Evaluation Program Implementation Plan	2
$PM_{2.5}$ Data Quality Objectives Process	2
FRM Performance Evaluation Program Field SOPs	3
QA Handbook, Vol. II, Part 1	3
40 CFR Part 50, Appendix L	4
40 CFR Part 58, Appendix A	4

1.5 Definitions

Appendix A contains a glossary of the terms in the PEP. Acronyms are in the front of the document.

1.6 Cautions

The filters used for the $PM_{2.5}$ sampler are comparatively small, each weighing about 150 milligrams (mg). Because of the size and weight of the particles that will be collected on these filters, net weights will be measured in micrograms (μg). The loads on the filter may be anywhere from 20 to 2000 μg (83 $\mu g/m^3$) with most sample loads around 300 μg. To put this weight in perspective, a 4-centimeter long human hair weighs ~312 μg. This 300 μg value represents ~0.2% of the weight of the blank filter. In addition, it is expected that the LA will be able to duplicate weighings of the same filter to within 15 μg. A single thumbprint on a filter weighs 15 μg. It should be apparent that any small loss or gain (i.e., finger oils, dust) will affect filter weights. Additional details of filter handling are discussed in Section 4.

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 2.0

General Laboratory Preparation

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: General Laboratory Preparation

SOP: PEPL-2.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP describes the process to select and set up a weighing and conditioning area within an environment designed to:

- minimize sources of contamination
- isolate filters and balance(s) from forces and environmental conditions affecting microbalance measurement stability and variability
- maintain the temperature of the conditioning air to a mean of 20 to 23 °C, controlled to ±2 °C over a 24-hour period
- maintain the relative humidity(RH) to a mean of 30 to 40%, controlled to ±5% RH over 24 hours
- maintain clean surfaces for exposing new filters, loading or unloading cassettes and storing filters when they are not being weighed or sent to or returned from the field
- in general, prepare the laboratory for activities required in the PEP

2.0 Summary of Method

The laboratory used for the conditioning and weighing of filters will have enough space to:

- condition the required number of filters
- house the microbalance, temperature and humidity recording instruments, bar code readers, and data recording devices (computer) in a manner that does not affect data quality
- distinguish filters at various stages of preparation and weighing
- prepare filters for shipping.

The room will meet temperature and humidity requirements, be kept free of sources of contamination, and be maintained in an acceptable state of cleanliness. This SOP assumes that the $PM_{2.5}$ filter laboratory has been established.

Acceptable control of conditions listed in 1.0 are determined by testing for and determining several parameters. Microbalance performance is estimated by determining the detection limit for small loads as compared to background noise and much larger filter weights.

The data for determining sensitivity by DL are simulated by measuring external standard weights which are more stable to weigh then the filters.

Balance stability is estimated by the time required to achieve stable standard weight values (10–20 seconds) and if the mean difference is less than 3 ug and the overall DL is less than or equal to 5 ug.

3.0 Definitions

Appendix A contains a glossary of terms used for the PEP. Acronyms are listed in the front of this document. However, the terms conditioning room and laboratory are used synonymously throughout this document.

4.0 Health and Safety Warnings

To prevent personal injury, employees must heed all warnings associated with operation of microbalances, and their supporting equipment and supplies. Specific health and safety warnings will generally be found at the point in the operating manual or troubleshooting guide where they are most applicable. In general, health and safety warnings will fall into these three categories:

- electrical,
- chemical, and
- equipment placement and stability.

Electrical safety considerations that apply to PM2.5 laboratory operations include the following:

- Make all electrical connections in accordance with national codes. Always use a third wire grounding arrangement on the microbalance and on any electrical appliances. To minimize the possibility of electrical shock and injury, always use a grounded outlet and cord. This process avoids the possibility of electrocution.
- Electrical supply lines to the $PM_{2.5}$ laboratory must be installed so that they remain dry under all weather conditions and are protected from exposure to extremes of light and heat, which will degrade the covering and insulation. Inspect the electrical cords and connections for signs of wear and have an electrician repair or replace them as needed.
- Always unplug the power to laboratory instruments when servicing or replacing parts in areas requiring removal of protective panels.

Chemical safety considerations that apply to $PM_{2.5}$ laboratory operations include the following:

- All chemicals and hazardous material used in the laboratory should come with material safety data sheets (MSDS). Post these, or have them in an area that is readily available.
- Use care in the application of cleaning solvents. Use of gloves is recommended. Wash hands thoroughly after working with chemicals. Provide good ventilation if organic solvents are used. Dispose of chemicals and shop towels properly.
- Mercury metal, a poisonous material, is present in some types of thermometers, barometers, and RH indicators. If liquid mercury is spilled, it must be cleaned up and disposed of properly. Use protective equipment to avoid inhalation of vapors and impermeable gloves to avoid skin contact. Mercury cleanup kits are available. Locate

mercury-containing devices in areas that are physically, structurally, and aerodynamically isolated from human workstations.

- Exercise caution when using antistatic devices containing radioactive polonium sources. Keep an inventory of the location and size of antistatic devices. Dispose of the devices in accordance with manufacturers specifications and State and local regulations.

Equipment safety considerations that apply to $PM_{2.5}$ laboratory operations include the following:

- Ensure that doors can open without banging into furniture equipment.
- Ensure that doors can open from the inside.
- Avoid placing shelves above head height or where personnel must reach over obstructions (e.g., desks) in a manner that they could and lose balance.
- Ensure that the design and performance of the weighing /conditioning room meet the facility Health and Safety Officer's minimum requirements for providing breathable air for employees entering and using the room. In general, the requirement can be met by an alarmed, feedback loop or equivalent throttle control of % of make-up(fresh air) which adds more fresh air when the CO_2 concentration rises above (2000ppm) and the oxygen concentration falls below the proportions considered acceptable for human respiratory needs. An example of such a make-up air throttle system is the inexpensive Gaztech® sensor/control made by Telaire.

The laboratory must be designed in a manner that alarm systems can be heard. Fire suppression equipment should comply with local building codes, State/Federal OSHA regulations and National Fire Codes.

5.0 Cautions

Relative humidity is a difficult parameter to control; even if total moisture content stays constant, if temperature changes RH will also change. Be prepared to control for seasonal extremes (such as high heat and humidity in the summer) by using supplemental equipment (i.e., as dehumidification in the summer) during the extremes.

Personnel should always wear clean clothes and wash thoroughly all parts of their body that may be exposed during weighing, especially hands, arms, face, and hair, using adequate soap and water to remove loose skin and hair, as close as possible to the weighing activity. The use of laboratory coats and gloves are required and will minimize the potential for laboratory contamination. Laboratory coats must be taken off before leaving the weighing facility to minimize contamination from the external environment.

6.0 Interferences

- An important limitation of the method involves changes in the weight of a collected sample due to mishandling, chemical reactions, and volatilization.

- Handling procedures, humidity and temperature control of the filter and sample during weighing, and promptness in and consistency of the weighing method prior to and following collection all help control artifacts.

- The chemical makeup of the $PM_{2.5}$ particulate matter will vary with the sampling location and the source. Thus, the magnitude of $PM_{2.5}$ weight changes due to chemical and physical processes will also vary with site location, day of the week, and season of the year.

- If nitric acid vapor is present at a sampling location, it can deposit on a Teflon® filter and cause small weight gains in proportion to the amount of nitric acid present in the atmosphere. This weight gain may not be controllable.

- Weight losses can occur due to thermal or chemical decomposition or evaporation of compounds like ammonium nitrate (NH_4NO_3), which releases ammonia and nitric acid as gases. Semivolatile organic compounds (SVOCs) may be part of the particulate matter on the filters; if so, they may evaporate and cause sample weight losses. Such weight losses will be minimized or standardized by keeping the filters cool during transport to the weighing laboratory and by conditioning and weighing the filters promptly after their receipt in the laboratory. The quantity of such changes will be made more comparable from filter to filter by the consistent performance of these procedures, including control of the environmental conditions.

- Some new blank Teflon® filters have been found to exhibit a weight loss of up to 150 micrograms (µg) over a period of time up to 6 weeks after they have been removed from their original shipping containers. Although it is anticipated that the filters used for the FRM PEs will not have this problem, analysts will check for weight loss in any new lot of filters that is received (see Section 6). Filters should not be used until their weights have stabilized.

- Weight loss due to mechanical removal of particles from the filter will be minimized by carefully removing the filter from its cassette, by conditioning the filter, and by neutralizing the electrostatic charge buildup on the filter before weighing.

- Weight gain or loss due to absorption or desorption of water vapor on the filter or on the particulate matter will be minimized by specifying low-moisture pickup for the filter media and by conditioning the filters within the specified humidity and temperature ranges, both before sampling and after receipt from the field.

- Filters may become contaminated during conditioning or weighing from particulate matter in the air, from dust on the microbalance or working surfaces, or from the LA or outside visitors(e.g., service technicians) who enter the laboratory inadequately prepared to prevent human-to-filter contamination. Airborne contamination will be reduced by passing the air inside the conditioning environment through a high-efficiency particulate air (HEPA) filter that is regularly changed. Surface contamination will be reduced by cleaning work surfaces with low-lint, disposable laboratory wipes before weighing the filters.

- Errors in the gravimetric analysis of samples can also result from the buildup of an electrostatic charge on the microbalance or on filters during their manufacture or sampling. This electrostatic charge buildup will interfere with the microbalance weighing. It can be reduced on the microbalance by electrically grounding the microbalance and coating the nonconductive surfaces with an antistatic solution. It can be reduced on filters by the use of polonium-210 (^{210}Po) antistatic strips immediately before the weighing process begins.

- Vibration, due to facility activity of equipment and personnel, may cause microbalance instability. Minimize the number of personnel in the weigh room during weighing activities, and schedule weighing activities when external influences to the microbalance are at a minimum. The use of balance pads and floor mats that reduce vibration may be necessary.

7.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training are required.

8.0 Apparatus and Materials

- 5, 100 and 200 mg ASTM Class 1 weights
- Forms GPL01
- microbalance
- nonmetallic, nonserrated weight forceps

9.0 Laboratory Preparation Procedures

9.1 Laboratory Control for Filter Measurement Stability

To ensure that the filter pre- and postsampling weight measurements will be as unbiased and precise as necessary, the following laboratory requirements for the weighing and conditioning environment have been established for the PM 2.5 PEP.

- **Mean temperature:** For the 24-hour period prior to a weighing session, the mean of the 5-minute averages for temperature must be a value from 20 to 23 °C ±2 °C,
- **Mean relative humidity:** For the 24-hour period prior to a weighing session the mean of the 5-minute averages for RH must be a value from 30 to 40% RH ±5% RH
- **Contamination:** laboratory blank weights must not fluctuate more than 15μg from the presampling and post-sampling weighing sessions.

NOTE: The tests for these requirements will be tested in SOP 6.01 and 8.01 as individual lot exposure blank tests.

9.2 Microbalance Stability and Sensitivity

The following summarizes the microbalance requirements established to ensure microbalance stability and sensitivity during and between pre- and postsampling measurements on the same filter:

- a readability and repeatability of at least ±1 µg
- operated with the leveling bubble at the level position
- automatically calibrated
- **not** automatically zeroed (for $PM_{2.5}$ mass weighing application)
- manual external verification of working standards according to the manufacturer's instructions
- internally calibrate (using the F1 key) according to the manufacturer’s instructions and this SOP at the beginning of each weighing session.
- located in a controlled environment
- located on a clean, vibration-free surface
- located in an environment free of air pulses or turbulence that might prolong or disrupt efforts to achieve stable weights
- the microbalance weighing chamber module grounded to a universal ground
- left plugged into power and with power on at all times (LCD screen can be left off)
- maintained and operated strictly according these SOPs and the manufacturer's instructions

Prior to implementing routine filter activities, establish that the laboratory can meet the microbalance requirements listed above. The LA will follow the procedures listed below:

1. Turn on the microbalance and let it warm up for 24 hours
.
2. Select the 5- 100- and 200- mg working standards.

3. Zero (using the TARE key) and calibrate (using the F1 key) the microbalance according to the microbalance's operating manual.

4. Open and close the microbalance's draft shield (circular arrow key) two times to equilibrate the air in the draft shield chamber with the conditioning room.

5. Using smooth, nonserrated, nonmetallic forceps, gently place each working mass reference standard on the sample pan. Close the microbalance's draft shield. Wait until the microbalance's display indicates that a stable reading has been obtained (~ 20 seconds after stable reading indicated) before recording a gravimetric measurement.

6. Record the measured value in mg to 3 decimal places on the Laboratory Balance Stability Test Form GLP-01.

7. Each hour repeat steps 1-6 until seven measurements of each working mass have been recorded.

8. For each concentration, calculate the difference between paired consecutive measurements and the mean difference of all seven and the mean and standard deviation of each standard weight.

9. For each standard, provide a detection limit by multiplying the standard deviation of each weight by 3 and entering it in the "DL" row.

10. Provide an overall detection limit by averaging the three detection limits and placing the value in the "Overall Detection Limit" row.

11. If the mean difference is less than 3 μg for each standard and the overall detection limit is less than 5 ug and the balance achieves a stable weight in ;less than 30 seconds, then balance stability has been demonstrated. If not, troubleshoot the microbalance system and the laboratory environment until stability is established.

10.0 Troubleshooting

The following section discusses laboratory equipment that may need troubleshooting.

Note: Keep a chronological record of all problems and corrective action for each piece of equipment or system. Attach copies of service technician reports and notes of any additional comments or observations made during the visit or in follow-up calls.

10.1.1 Conditioning Sensor and Control Equipment

See the Troubleshooting section of the manufacturer's manual for the environmental control and monitoring system. There may need to be some additional control of air flow patterns. See Appendix C for additional information and tests for this problem .

If the laboratory conditioning system is cycling on and off so frequently that it disrupts weighing by causing fluctuations in weight that take more than approximately 20 to 30 seconds to stabilize, take the steps described in the following 2 paragraphs.

Ensure that the entrance (door) to the conditioned area is closed completely. Check temperature and RH, using continuous recording and readouts to determine if the value of one or the other is climbing outside of the acceptable set-point limits when the rise starts to reverse itself. Check the particulate filter to determine if it has become overloaded and needs to be changed or if the filter opening has accidentally been partly or completely obstructed. If cooling air should be coming from the air inlet into the room, check the temperature of the air to see if it is cooler or warmer than expected (as usual for the conditions).

If the temperature is rising to the set point and then falling rapidly, ask the facility engineer to determine the cause and try to correct it with existing installed equipment. If a service contract has been arranged for maintaining the environmental control system within the required set points, then the LA should request the authorized person (through the WAM) to visit. The LA should check the temperature sensor and verify that it is responding accurately. If it is, then the service technician can check the control mechanism to determine if it is faulty. If not, the flow rate may be increased by the service technician to bring more cool air in faster. If such a change is made, the technician should check to see that the desired air pressure gradients and the RH are not altered. This "tweaking" may have to occur a number of times until all the major different environmental condition combinations that occur locally during a period of 6 months to 1 year have been addressed by specific system adjustments.

10.1.2 Microbalance

See pages 1-43 and 1-44 of the troubleshooting guide of the Sartorius MC5 Installation and Operating Instructions.

Check that the weight reading is outside of the limit of acceptable fluctuation (3 μg). If the steps involve diagnoses or adjustments to be performed by the service technician, ask the WAM to call the service technician.

If the presampling filter weights are outside acceptance requirements, check to confirm that no visible contamination has been adsorbed onto the filter. If none is found, check the shelf-life (half-life) of the polonium strips being used to control buildup of static charge.

10.1.3 Conditioning Room

If lab blanks in the conditioning room gain weight over a time period that is short enough to be considered significant, laboratory contamination may be a problem.. If the conditioning/weighing room contains equipment and/or is used by more than two people at a time or for more than $PM_{2.5}$ sample filter conditioning and weighing operations (at a time or at all), reduce the potential for contamination and other weighing impacts.

Test to see that the air pressure gradient is higher at the balance and conditioning areas than in the rest of the room and higher in the room than just outside the entrance(s) to the room. The air pressure gradients and air flow patterns should be adjusted to constantly move any suspended particles away from the balance and conditioning areas and thereby prevent intrusion of particles.

Change work activity patterns (by reducing them if they have been occurring during or just before conditioning or weighing). Try alternative protective clothing and cleaning procedures. For example, if you are not using sticky mats and booties, try using them. If you are using any cleaning sprays, stop.

10.1.4 Filters

At certain times, all field (or loaded lab) sample filters used as duplicate or replicate QC checks vary in weight more than 30 μg in a week, even though temperature and RH are within the required control limits and the age of the polonium strips is still within the half-life.
A corona discharge type of “clean air “generator can be used to determine if the problem can be solved.

11. Data Acquisition and Calculations

10.1 Data Acquisition

Test form GLP-01 will be used to collect the appropriate information to establish laboratory control.

10.2 Calculations

The following calculations will be used in Form GLP-01:

Difference for a single check *(d)* - The difference between each filter value will be calculated. The difference, *d*, for each check is calculated using Equation 1, where *X* represents the concentration produced from the original weight (day 1) and *Y* represents the concentration reported for the second weight (day 1+1).

$$d = |Y - X| \qquad (2\text{-}1)$$

Mean (d_z) - The mean is used to determine the means of the 24-hour temperature and RH and the mean differences of each filter value. Mean d_z, for each particular variable within the 5-day test, is calculated using equation 2, where d_1 through d_n represent individual values making up the mean and *n* represents the number of values (i.e., 5) in the test.

$$d_z = \frac{d_1 + d_2 + d_3 \ldots\ldots d_n}{n} \qquad (2\text{-}2)$$

Standard Deviation (SD)- This equation will be used to determine the Standard Deviation (SD) of the 5-minute temperature and RH values. These should be provided by the automated temperature and humidity programs and will use Equation 3, where d_i is each individual value, d_z is the mean established in Equation 2, and *n* is the number of values.

$$SD = \sqrt{\frac{\sum (d_i - d_z)^2}{n - 1}} \quad (2\text{-}3)$$

Detection Limit- The detection limit (DL) will be used to determine when a measured value becomes believable because it is larger than the uncertainty associated with it. The point when this occurs is defined as the limit of detection and is calculated using Equation 4.

$$DL = SD * 3 \qquad (2\text{-}4)$$

Balance Stability Test						
Run	5 mg Standard **ST1**	Difference	100 mg Standard **ST2**	Difference	200 mg Standard **ST3**	Difference
1						
2						
3						
4						
5						
6						
7						
Mean						
SD						
DL						
Overall Detection Limit =						
GLP-01						

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 3.0 Equipment Inventory, Procurement, Receiving and Maintenance

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Equipment Inventory, Procurement, Receiving, and Maintenance

SOP: PEPL-3.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP explains the procedures involved with inventorying existing laboratory equipment, receiving new equipment and consumables, and maintaining the equipment.

2.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

3.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training is required.

4.0 Apparatus and Materials

The LA will use the following apparatus and materials to perform the procedures in this section:

- Table 3-1 provides a listing of the equipment and consumables needed for the laboratory. The LA will be responsible for ensuring that all equipment and consumables on this list are present.
- Laboratory Inventory Form INV-01.
- Laboratory Procurement Log Form PRO-01.
- Laboratory Receiving Report Form REC-01.
- Table 3-2contains the laboratory maintenance schedule.
- Laboratory Maintenance Report Form MAN-01.
- Laboratory Maintenance/Service Report Form MAN-02.

5.0 Procedures

5.1 Equipment Inventory

During the summer of 1998, Regions 4 and 10 started developing laboratory capabilities for the $PM_{2.5}$ PEP weighing facilities. Table 3-1 provides a list of the capital equipment and consumables required for the mass weighing activities. The list, in many cases, identifies a suggested source and does not necessarily represent the equipment available at each facility. The LA will follow the procedure below:

1. Select Laboratory Inventory Form INV-01.

2. Take a complete inventory of all equipment and supplies.

3. Keep an original copy and file under Aerometric Information Retrieval AIRP/486. Provide a copy of the inventory to the WAM.

NOTE: Filter inventory is discussed in PEPL SOP 5.01, subsection 7.2.1.

The LA should maintain a 2- months' supply of consumables. During the first weeks of implementation, the LA will determine how quickly he/she is using consumable equipment and develop a purchasing schedule to ensure an adequate supply is maintained.

Table 3-1. Equipment/Consumable Inventory

Quant.	Units	Item	Vendor	Model Number
2	each	Microbalance	Sartorious	MC-5
2	set	ASTM Class 1 weight set	Rice Lake Weighing Systems	11909
2	each	Balance table	Fisher- Scientific	HM019945
2	each	Computer	Dell	
2	each	Bar code reader		
1	each	Bar code printing software		
1	each	Humidity/temp monitor	Visala	E-37510-02
1	each	Humidity/temp standard	Fisher -Scientific	11-661-78
1	each	NIST traceable thermometer	Fisher-Scientific	15-041A
1	each	Tacky mat plastic frame	Fisher-Scientific	06-528A
1	each	Uninteruptable power supply	Cole-Parmer	E-05158-60
1	each	Refrigerator		
1	each	Freezer		
1	each	Dishwasher		
2	each	Antifatigue floor mat	Richmond	19-61-763
2	each	Equilibration rack		
1	each	Laser jet printer		
1	each	De-humidifier		
1	each	Light table		
1	each	Microsoft® Access 97 Win 32		077-00370
		Sarto-Wedge®software for	Sartorius	YSW01
		Bar code printing software	Cole-Palmer	E-21190-10
24	each	HVAC filters		
1	case of 1,000	Powder-free antistatic gloves	Fisher-Scientific	11-393-85A
12	each	Polonium strips	NRD	2U500
7	pack of 100	Petri-dish slides	Gelman	7231
1	case of 12 bottles	Staticide	Cole-Parmer	E-33672-00
1	case of 15 packs	Low-lint wipes	Kimwipes	34155

Quant.	Units	Item	Vendor	Model Number
1	each	HVAC service contract	Local	
1	each	Microbalance service contract (2 scheduled visits per year)	Sartorius	
6	sets	Chart paper & pens		
1		Cleaning supplies	Local	
2	each	Worklon antistatic lab coats	Fisher-Scientific	01-352-69B
2	each	Forceps (SS w/plastic tips)	VWR	25672-100
1	case	Antistatic 3"x5" reclosable bags (for cassettes)	Consolidated Plastics	90202KH
1	box	Barcode stickers		
1	case of 1,000	Alcohol swipes	Fisher-Scientific	14-819-2
20	each	6-Pack coolers		
4	case of 24	Reusable U-Tek refrigerant packs (-1C)	Fisher-Scientific	03-528B
1	case	Antistatic 9 X12" reclosable bags (for data sheet)	Consolidated Plastics	90210KH
4	each	Log books		
20	each	Min/max thermometers (various digital ones available)	Sentry	4121
3	120 sheets	Hard surface tacky mat (moderate tack)	Fisher-Scientific	06-527-2

5.2 Procurement

As consumables run low or new equipment purchases are necessary, the LA will be responsible for assisting in the procurement of these items following the policy and requirements described in the ESAT scope of work. The LA should continue purchasing consumable equipment with the same model numbers as initially procured unless the WAM suggests a different item due to improved quality, reduction in contamination, ease of use, or lower cost (without sacrificing quality). The following procedures will be required:

1. The LA will develop procurement requests based on EPA requirements.

2. Upon order, add items to Laboratory Procurement Log Form PRO-01.

3. Once a month provide a copy of the Form PRO-01 to the WAM.

4. File Form PRO-01 in file AIRP/486.

5.3 Receipt

Filter receipt is discussed in PEPL SOP 5.01 . Upon receiving equipment and consumables, the LA will take the following steps:

1. Pull the appropriate purchase order for the incoming items from the files.

2. Fill out Laboratory Receiving Report Form REC-01 comparing the items and quantity against the purchase order and inspecting the condition of each item.

3. If the items received match the purchase order and the condition of the equipment or consumables is acceptable, signify this on the form and file it in AIRP/486.

4. If the quantity, items, or conditions are not acceptable, complete Form REC-01 with remarks and send a copy of the form to the WAM.

5. Add receipt information to the Laboratory Procurement Log Form PRO-01

5.4 Maintenance

Maintenance is a program of actions to ensure that the laboratory maintains the appropriate conditioning environment, maintains contamination at acceptable levels, and prevents equipment from failing during use. Table 3-2 provides a listing of the required maintenance activities, the personnel responsible for this maintenance, and the frequency of each activity.

Table 3-2. Maintenance Requirements

Item	Responsibility	Service Agreement # (if appropriate)	Frequency
General lab maintenance			
Cleaning			
Table cleaning	LA		Every day
Overall lab	LA		Once a month
Cassette ethanol wiping/washing	LA		After each use
Adhesive-coated floor mats	LA		Weekly or when soiled to a point of non-performance
HEPA filter change	LA		Once a month
Polonium strip change	LA		Every 6 months
Polonium strip cleaning	LA		Monthly or as shown by blank data
Microbalance			
Cleaning	LA		6 months
Service cleaning/calibration	Service provider		Twice a year
Calibration verification	LA		Every sample weighing
Temperature/humidity readers			
Calibration Verification	LA		Once every 3 months

5.4.1 General Laboratory Maintenance

5.4.1.1 Daily Cleaning

1. Every day that the laboratory is in use, the tables used to perform laboratory activities should be carefully wiped down with a damp, lint-free Kimwipe.

2. Use a separate Kimwipe dampened with ethanol to clean the forceps used to move the metal standard weights and those used to handle the filters.

5.4.1.2 After Each Use Cleaning

Cassettes - After filters have been removed for conditioning (PEPL-10.01)

1. Remove the protective (metal) cap from the cassette and verify its cleanliness. If required, wipe the inside of the cap with a Kimwipe dampened with ethanol.

2. Check the condition and cleanliness of the outside and inside of the cassette top and bottom and backing screen. Place all cassettes and screens in a dishwasher. Wash one cycle with deionized water.

5.4.1.3 Monthly Cleaning

Laboratory

On the selected cleaning day, have no exposed filters and as few other filters in the laboratory as possible. Do not use vacuums for cleaning.

1. Place Petrislide tops on all filters that are exposed in the conditioning environment.

2. Place the filters in a storage container, close the lid, and place the container in an area that is least affected by the cleaning activity.

3. Wipe down all surfaces with a clean, damp, lint-free Kimwipes, including the large baggies containing items on the supplies and filter conditioning shelf in the chamber.

4. Mop the floor with a damp mop. Remove as much excess water as possible to reduce relative humidity fluctuations.

5. Change the HEPA filter.

6. Upon completion of maintenance, fill out Laboratory Maintenance Activity Report Form MAN-01.

Antistatic Polonium Ionizing Strips

1. Remove the antistatic, ionizing polonium strips from their mounting (for example, from the gooseneck, balance chamber, or other holder).

2. Clean and deionize the inside of the mounting cavities. For the inside of a balance cavity or chamber, use a cottton-tipped applicator wetted with household ammonia. Wipe with a second cottton-tipped applicator wetted with water and let air-dry. Use extra caution and care. Gently brush the exposed surfaces of the cavity. Gently close the chamber door and gently brush the outer surface with the ammonia tip to control static charge.

3. Clean the top surface and the strips of the antistatic ionizing units by gently rubbing with a Kimwipe applicator wetted with ethanol.

4. Replace the ionizing unit in the weighing chamber and gooseneck holders.

5.4.2 Microbalance Maintenance

5.4.2.1 Calibration

ASTM Class 1 mass reference standards must be used for the microbalance calibration.

1. The microbalance will be calibrated on a regular basis (e.g., twice yearly) by an authorized microbalance service technician and maintained according to the manufacturer's recommendations. The calibration will be traceable to the National Institute of Standards and Technology.

2. If the microbalance is found to be out of calibration during routine weighing operations, recalibrate it according to the instructions given in SOP PEPL-6.02. If the microbalance cannot be calibrated, request that it be serviced by an authorized microbalance service representative. Do not attempt to adjust or repair the microbalance.

5.4.2.2 Cleaning

Clean the microbalance housing and the draft shield twice a year or when contamination may be indicated using the following steps:

1. Unplug the power supply from the wall outlet before cleaning the microbalance.

2. Use a piece of lint-free cloth that has been wetted with ethanol to clean the weighing chamber. Ensure that no liquid enters the microbalance housing.

3. Wipe down the microbalance after cleaning with a soft, dry piece of cloth.

4. Remove the draft shield from the weighing cell. Cover the exposed weighing cell area (left exposed by draft shield) with aluminum foil to prevent contamination.

5. Clean the draft shield with a commercially available glass cleaning agent or in a dishwasher.

6. Coat the draft shield with antistatic solution.

NOTE: Do not insert forceps or any other object behind the draft shield closing plate. The weighing system is hermetically sealed from the draft shield closing plate so that dirt cannot enter.

5.4.3 Servicing

Laboratories may enter into various service agreements to ensure that laboratory equipment and facilities operate properly. Some of these agreements will include scheduled maintenance activities, and others may be set up for servicing during a malfunction. Frequent checks on the instrument should indicate whether servicing is required. If data indicate equipment servicing, use the following procedures:

1. Fill out Laboratory Maintenance/Service Report Form MAN-02 and send to the WAM; the WAM will contact the service provider.

2. Upon servicing, complete MAN-02. Query service provider on what was accomplished.

3. The LA and the service provider should sign Form MAN-02

4. Keep the original and file it in AIRP/486. Distribute a copy to the WAM.

5. Place appropriate information from form MAN02 onto Laboratory Maintenance Activity Report Form MAN-01.

NOTE: The LA should also record routine servicing on Laboratory Maintenance/Service Report Form MAN-02.

Laboratory Inventory Form (INV-01)					
Item	**Vendor**	**Model #**	**Quantity**	**Purchase Date**	**Warranty**
Capital Equipment					
Microbalance					
ASTM Class 1 weight set					
Balance table					
Computer					
Bar coder Reader					
Bar code printing Software					
Humidity/temp monitor					
NIST traceable thermometer					
Sling psychrometer					
Tacky mat plastic frame					
Uninteruptable power supply					
Refrigerator					
Freezer					
Dishwasher					
Antifatigue floor mat,					
Acrylic desiccator with sliding tray's					
Laser jet printer					
De-humidifier					
Light table					
Microsoft® Access 97 Win 32					
Sarto-Wedge®software					
Bar code printing software					
Software for temp and RH data logger					
Microbalance service Contract (2 scheduled visits per year)					
Worklon antistatic lab coats					
Forceps (SS w/plastic tips)					

Laboratory Inventory Form (INV-01)					
Item	**Vendor**	**Model #**	**Quantity**	**Purchase Date**	**Warranty**
Consumable Equipment					
Chart paper & pens					
Cleaning supplies					
Antistatic 3"x5" reclosable bags (for cassettes)					
Bar-code stickers					
HVAC filters					
Powder-free antistatic gloves					
Polonium strips					
Petri-dishes					
Staticide					
Low-lint wipes					
HVAC service contract					
Form INV-01					

Laboratory Procurement Log

Item	Model #	Qty	PO #	Vendor	Date		Cost	Initials	Accept/ Reject
					Ordered	Received			

Form PRO-01

Laboratory Equipment/Consumable Receiving Report

Date:_____

Received From:				
Shipped From:				
Shipped Via:				
Shipping Charge	Prepaid	Collect	Freight Bill #	
Purchase Order Number				
Quantity	Description Of Item			Condition
Remarks: Accept Shipment _____ Problem_____				
Notes:				
Form REC-01				

Laboratory Maintenance Activity Report		
Activity	Date	Initials
Form MAN-01		

Laboratory Maintenance/Service Report		
Item Name:	Time:	Date:
Manufacturer:	Model:	
Failure Condition		
Location:	Interval since last service:	
General Description of Failure and Cause		
Corrective Action Taken		
Failed Components Replaced		
Failed Components Repaired		
Service Requirements		
Preventive Maintenance Completed		
Operational Test Completed		
LA Signature **Form MAN-02**	Service Provider Signature	

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 4.0 Communication

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Communication

SOP: PEPL-4.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP describes the required activities for PEP laboratory personnel to communicate technical information to organizations intimately involved in the program and includes

- ESAT WAM for the LA,
- ESAT WAMs for the Field Region,
- ESAT Field personnel, and
- OAQPS.

This SOP does not describe additional ESAT communication obligations described in the ESAT Scope of Work(SOW). Communications include reports, e-mail messages, and phone calls.

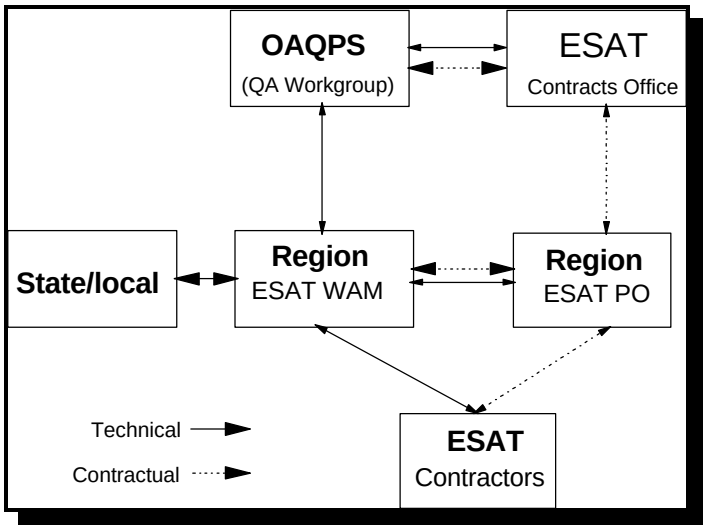

Figure 4.1 Line of communication

2.0 Summary of Method

An organized communications framework facilitates the flow of information among the participating organizations as well as other users of the information produced by the $PM_{2.5}$ network. Figure 4.1 represents the principal communication pathways. In general, ESAT contractors will be responsible for informing Regional WAMs and Project Officers (POs) on technical progress, issues, and contractual obligations. On the technical side, EPA Regional WAMs will be responsible for communicating with State and local agencies and informing OAQPS about issues that require technical attention. Contractual issues will be conveyed from the ESAT contractor through POs to the ESAT Contracts Office and, if necessary, to OAQPS. Table 4.1 at the end of this SOP lists important EPA ESAT contacts.

The ESAT contractors will have frequent communication with Regional WAMs on the progress of their activities and any problems/issues associated with them. Resolution of these issues should take place in the Regions unless the issue could affect the implementation of the program at a national level. In those cases, it can be discussed and resolved through the ESAT Workgroup conference call.

3.0 Definitions

3.1 Acronyms

Acronyms and abbreviations are listed in the front of this document.

3.2 Forms

The following forms are found at the end of this SOP:

COM-1 -- Phone Communication Form,
COM-2 -- Weekly Progress Report, and
COC-1 -- Filter Inventory and Tracking Form.

4.0 Apparatus and Materials

The following capital/consumable equipment will be required for communications:

- telephone
- laboratory personal computer with Internet and EPA e-mail capabilities
- laser jet printer
- writing utensils
- laboratory communications notebook
- appropriate forms

5.0 Communication Procedures

5.1 Phone Communications

5.1.1 Issue-Related Calls

A call may be initiated by the WAM(s) field personnel or by the LA at any time during implementation. During the conversation, Phone Communication Form(COM-1), in the laboratory communications notebook, will be used by the LA to record the conversation. Notes will include the following:

- date
- time
- personnel involved
- issue(s)
- decision(s)
- follow-up action(s)

- follow-up action responsibility
- follow-up action completed by (date)

If follow up action is required by the LA, these actions will be included in the weekly progress reports (see sub-section 5.2 of this SOP). At a minimum, the LA will keep the original hardcopy in the laboratory communications notebook. He/she may also want to keep an electronic record of this information on diskette.

5.1.2 Field Communication

5.1.2.1 Filter Shipment

Every 2 weeks, filters will be shipped to the 4 field regions by Federal Express (see Section 8) next-day mail and hand carried to the field scientists working in Regions 4 or 10. On the day of shipment, the LA will communicate with the field scientist (see Table 4-1, the field scientist list) and provide the following information:

- date of shipment
- number of filters in shipment
- number of boxes in shipment
- air bill number

The LA will also send the field scientist an e-mail containing the same information and will carbon copy both their ESAT WAM and the EPA Regional Field WAM (See Table 4-1).

5.1.2.2 Equipment Shipment

Once a month, the laboratory will ship coolers, max/min thermometers, and ice substitutes back to the regional offices by the United Parcel Service(UPS) or FED Ex (see PEPL-9.01). On the day of shipment, the LA will communicate with the field contact (see the field contact list) and provide the following information:

- date of shipment
- number of boxes in shipment
- tracking number

The LA will also send the field contact an e-mail containing the same information and will carbon copy both their ESAT WAM and the EPA Regional Field WAM (See Table 4-1).

5.1.2.3 ESAT Conference Calls

LAs may be asked to participate on ESAT Workgroup conference calls to discuss progress or resolve issues. WAMs will inform the LA of information that needs to be prepared for the call at

least 3 days prior to the call. During the call, the LA will use Phone Communication Form COM-1 to record issues/action items that pertain to their laboratory. These items will be included in the next weekly progress report.

5.2 Weekly Progress Reports

The LA will provide the WAM a progress report in writing every Friday or on the last day of the scheduled work week. Weekly Progress Report Form COM-2 will be used to convey the following information:

- Reporting date - beginning and ending date that report covers
- Reporter - person writing reports
- Progress - progress on laboratory activities
  - Presampling processing- filters prepared within reporting date
  - Postsampling processing- filters weighed within reporting date and data submitted to AIRS
  - Shipments- shipments made to each Region within reporting date
  - Receipt - filters received within reporting date (totals)
- Issues -
  - Old issues- issues reported in earlier reports that have not been resolved
  - New issues- issues arising within reporting date
- Actions - action necessary to resolve issues including the person(s) responsible for resolving them and the anticipated dates when they will be resolved.

In addition, and updated Filter Inventory and Tracking Form COC-1 will be included with the weekly progress report. The LA will maintain a complete record of the weekly progress report in a three-ring binder.

5.2.1 Filter Inventory and Tracking Form

Filters must be used and weighed within prescribed time periods (see Figure 4.2). These time periods should be checked. The laboratory will track filters from pre-sample weighing to AIRS upload using Filter Inventory and Tracking Form COC-1. All filters that were preweighed should be placed on this tracking form. If filters are voided for some reason during the data collection process, a flag should be included on the form. The Filter Inventory and Tracking Form COC-1 ensures that the filter time periods are met as well as indicates the stage of operation a filter is undergoing. Based upon the concepts for the data management system (see Section 11), the information on this form will be included on other data entry screens; therefore, the Filter Inventory and Tracking Form COC-1 may be a reporting feature.

5.3 Communication Summary

Table 4-1 provide a summary of the major communications activities.

Table 4-1 Communications Summary

Person	Communicates to	Communication Function
Lab WAM	OAQPS LA	Bulk filter shipments Additional resources needs Review of deliverables Review of data Corrective action Schedule changes
LA	WAM FS OAQPS	Lab progress Problems/issues/schedule changes Outgoing filter/equipment shipment Filter shipment receipt from field AIRS uploads
FS	LA	Filter shipment from field Electronic mailing of field data Filter/equipment requests
OAQPS	LA	Data transferred to AIRS

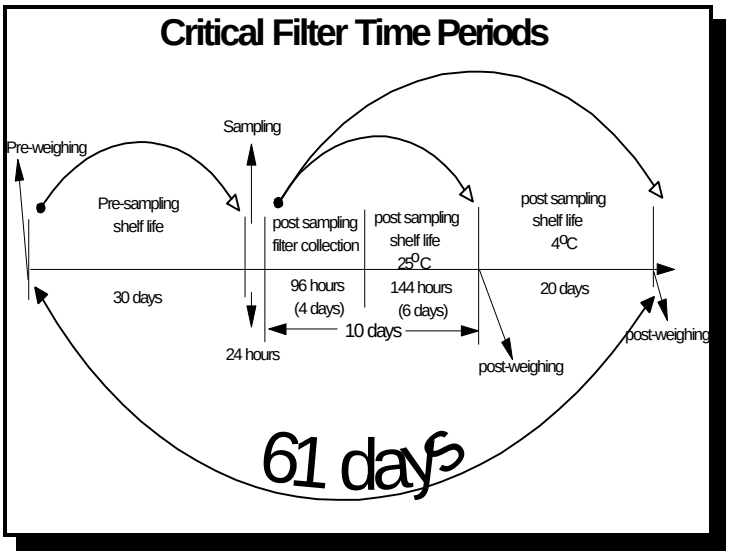

Figure 4.2 Critical filter holding times

5.4 Laboratory Time Lines

One aspect of the implementation process that is time critical is the filter holding time dates. As is illustrated in Figure 4.2 and stipulated in the Code of Federal Regulations, filters must be used within 30 days of pre-sampling weighing or they must be reconditioned and pre-weighed. Figure 4.2 indicates that filters must be collected within 96 hours of the end of the sample period. In most instances the FS will collect the filters within 8 to 48 hours of the end of the sample period. Samples will be sent the day of removal to the appropriate laboratory via next day delivery. Data will be immediately downloaded from the portable sampler and stored in two mediums (hard drive and two diskettes). One diskette of the data will be shipped with the sample. Data may also be transmitted, via modem, to the appropriate laboratory. Table 4-2 provides a summary of the key activities discussed above.

Table 4-2 Implementation Summary

Implementation Phase	Activity	Acceptable Time frame
Laboratory	Pre-sampling weighing	30 days
	Post-sample weighing	10 days
	Data input/review/validation	10 working days
	AIRS Upload	5 working days
Field	Filter use	30 days of pre-sample weighing
	Filter collection	8-48 hours from sample end date/time
	Filter/data shipment	within 8 hours of sample removal

Table 4-3 provides an estimate of the number of filters to be prepared for the field each month (filters/month); it includes field blanks and collocated filters but does not include laboratory QC filters. This spread sheet was developed for the Region 4 and 10 laboratories to help provide a more accurate estimate of filter preparation. This estimate is based upon the numbers of SLAMS/NAMS samplers that are expected to be sited in FY 98. However, the actual values may be somewhat higher when additional information on the exact method designations for each routine monitor that each reporting organization within a Region will receive.

Table 4-3 Filter Estimates

Region	NAM/SLAMS	sites/year	sites/quarter	site/month	filters/month	filters/year
1	67	17	17	6	9	113
2	58	15	15	5	8	99
3	95	24	24	8	13	155
4	181	45	45	15	24	284
5	162	41	41	14	21	255
6	114	29	29	10	15	183
7	66	17	17	6	9	111
8	51	13	13	4	7	89
9	105	26	26	9	14	170
10	48	12	12	4	7	84
Total	**947**	**239**	**239**	**81**	**127**	**1575**

Based upon the estimates in Table 4-3, Table 4-4 provides a summary of the monthly filter preparation requirements for each laboratory.

Table 4-4 Monthly Filter Preparation Estimates.

Region 4 Laboratory		**Region 10 Laboratory**	
Region	Monthly Filter Requirement	Region	Monthly Filter Requirement
1	9	5	21
2	8	7	9
3	13	8	7
4	24	9	14
6	15	10	7
Total	**69**	**Total**	**58**

Figure 4.2 also indicates that filters must be weighed within 10 days (if maintained at 25ºC) or 30 days (if maintained at 4ºC) of the sampling end date. The Region 4 and 10 laboratories will be able to post-sampling weigh within 10 days of sample receipt, even though they will maintain filters at 4ºC prior to filter conditioning.

6.0 Records Management

Weekly progress reports will be archived in the laboratory reporting package file under AIRP/484. Phone communications will be archived in the laboratory reporting package file under SAMP/502/COM. See Section 13 for details.

Phone Communication Form (COM-1)		
Date:	**Time:**	**Recorder:**
Personnel on call:		
Issue(s):		
Decisions(s):		
Follow-up Action(s):		
Follow up Responsibilities:		
Completion Dates for Follow up Actions:		

Weekly Progress Report (COM-2)	
Reporting Date: Start: End:	**Reporter**:
Progress	
Presampling Processing: Filter weighed this period:	**Postsampling Processing:** Filters weighed this period: Filter data uploaded to AIRS:
Filter Shipments: Region Filters Date	**Filter Receipt: Region Filters Date**
Issues	
Old:	**New:**
Actions:	**Actions:**
Free-Form Notes:	

Table 4-5. ESAT Contacts

Name	Address	Phone Number	Electronic Mail
ESAT			
Angela Edwards Kathleen Engel Monica McEaddy Colleen Walling	U.S. EPA 401 M Street, SW Washington, DC 20460 Monica and Colleen Walling 5203G Kathleen and Angie 3805R	(703) 603-8709 (202) 564-4504 (202) 564-4503	edwards.angela@epa.gov engel.kathleen@epa.gov mckeaddy.monica@epa.gov walling.colleen@epa.gov
OAQPS			
Michael Papp Tim Hanley Mark Shanis	U.S. EPA Office of Air Quality Planning & Standards MQAG (MD-14) RTP, NC 27711	(919) 541-2408 (919) 541-4417 (919) 541-1323 (919) 541-0528	papp.michael@epa.gov hanley.tim@epa.gov shanis.mark@epa.gov
REGIONS			
Region 1 **WAM** Mary Jane Cuzzupe **PO** Tony Palermo **FS**	U.S. EPA Region 1 New England Regional Laboratory 60 Westview Street/EMALEX Lexington, MA 02173	 (781) 860-4383 (781) 860-4682	 cuzzupe.maryjane@epa.gov palermo.anthony@epa.gov
Region 2 **WAM** Clinton Cusick **PO** Dick Coleates **FS**	U.S. EPA Region 2 Raritan Depot/MS103 2890 Woodbridge Ave. Edison, NJ 08837-3679	 (908) 321-6881 (732) 321-6662	 cusick.clinton@epa.gov coleates.dick@epa.gov
Region 3 **WAM** Theodore Erdman **PO** Fred Foreman **FS**	U.S. EPA Region 3 841 Chestnut Building/3ES11 Philadelphia, PA 19107 U.S. EPA Region 3 Office of Analytical Services/3ES-20 839 Bestgate Road Annapolis, MD 21401-3013	 (215) 597-1193 (215) 566-2766	 erdman.ted@epa.gov foreman.fred@epa.gov

Name	Address	Phone Number	Electronic Mail
Region 4 **WAM** Herb Barden Steve Hall	U.S. EPA Reg 4 Science and Ecosystem Support Division 980 College Station Road Athens, GA 30605-2720	 (706) 355-8737 (706) 355-8615	 barden.herbert@epa.gov hall.johns@epa.gov
PO Mike Birch	U.S. EPA Region 4 APTMD Atlanta Federal Center 61 Forsyth St., SW Atlanta, GA 30303-3104	(706) 355-8552	birch.mike@epa.gov
FS			
LA			
Region 5 **WAM** Gordon Jones	U.S. EPA Region 5 77 West Jackson Blvd./AR18J Chicago, IL 60604-3507	 (312) 353-3115	 jones.gordon@epa.gov
PO Jay Thakkar	/ SM5J	(312) 886-1972	thakkar.jay@epa.gov
FS			
Region 6 **WAM** Kuenja Chung	U.S. EPA Region 6 First Interstate Bank Tower at Fountain Place 1445 Ross Avenue Dallas, TX 75202-2733	 (214) 665-2729	 chung.kuenja@epa.gov
PO Melvin Ritter	U.S. EPA Region 6 Laboratory Houston Branch/6MD-HC 10625 Fallstone Road Houston TX 77099	(281) 983-2146	ritter.melvin@epa.gov
FS			
Region 7 **WAM** Mike Davis	U.S. EPA Region 7 ENSV/EMWC 25 Funston Road Kansas City, KS 66115	 (913) 551-5081	 davis.michale@epa.gov
PO Harold Brown	U.S. EPA Region 7 726 Minnesota Ave/ENSV/RLAB Kansas City, KS 66101	(913) 551-5127	brown.harold@epa.gov
FS			
Region 8 **WAM** Joe Delwiche	U.S. EPA Region 8 999 18th Street/8P2-A Suite #500 Denver, CO 80202-2466	 (303) 312-6448	 delwiche.joseph@epa.gov
PO Barbara Daboll	/8TMS-L	(303) 236-5057	daboll.barbara@epa.gov
FS			

Name	Address	Phone Number	Electronic Mail
Region 9 **WAM** Mathew Plate . **PO** Rose Fong **FS**	U.S. EPA Region 9 75 Hawthorne St./PMD-3 San Francisco, CA 94105	(415) 744-1493 (415) 744-1534	plate.mathew@epa.gov fong.rose@epa.gov
Region 10 **WAM** Karen Marasigan **PO** Gerald Dodo **FS** **LA**	U.S. EPA Region 10 1200 Sixth Ave/ES-095 Seattle, WA 98101 U.S. EPA Region 10 Manchester Laboraory 7411 Beach Drive East Port Orchard, WA 98366	(206) 553-1792 (206) 553-8728	marasigan.karen@epa.gov dodo-gerald@epa.gov

** OAQPS AIRS Data Base Manager

Filter Inventory and Tracking Form

Filter ID	Cassette ID	Presample Weigh Date	Pre-sample Shipping Date	Region	Sample Start Date	Sample end Date	Laboratory Receipt Date	Postsample Weigh Date	AIR upload Date	Flags	LA Initials
FORM COC-1											

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 5.0 Filter Handling, Inventory, and Inspection

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Filter Handling, Inventory, and Inspection

SOP: PEPL-5.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP provides general guidelines for handling filters and a method for receiving, inspecting, and accepting filters.

2.0 Summary of Method

Unexposed filters will be received from OAQPS; inventoried, and inspected for quality prior to filter conditioning.

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

4.0 Cautions

Due to the size and amount of particulate matter that are expected on these filters, filter handling can be a major source of error in the laboratory. Exercise care in handling both unexposed and exposed filters. Strictly follow all procedures concerning weighing, labeling, and transporting filters to reduce the chance for measurement errors. Avoid rough handling of exposed filters because this may dislodge collected particulate matter on the filters. Avoid inadequate conditioning of filters or excessive delays between sample retrieval and sample weighing because this may lead to positive or negative weight changes and, thus, to inaccurate $PM_{2.5}$ concentrations.

5.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training is required.

6.0 Apparatus and Materials

The LA will use the following apparatus and materials to perform this procedure:

- filters
- filter inventory/inspection Form INV-02,
- light table
- Forceps, smooth non-serrated, stored in a plastic bag
- laboratory coat
- Powder-free, antistatic gloves
- low lint laboratory wipes
- Petrislides

7.0 Procedures

7.1 Filter Handling

The goal of filter handling is to perform the necessary work without contaminating the filters. SOPs in other sections contain details of filter handling procedures at the appropriate stages of the laboratory process. However, the following are important filter-handling points:

- wear powder free antistatic gloves and a laboratory coat whenever handling filters. Gloves that are packed in a box can carry an electrostatic charge. Discharge this charge by touching a good electrical ground with the back of the hand after putting on the gloves.
- use clean, smooth, nonserrated forceps. Mark these forceps to distinguish them from the forceps used to handle mass reference standards.
- carefully handle the filters with the forceps, holding the support ring, rather than the filter media
- clean the forceps with low lint disposable laboratory wipes and then allow them to air-dry before handling filters
- keep forceps in a plastic bag when not in use
- clean the forceps with low lint disposable laboratory wipes if they ever touch the filter material of an exposed filter to avoid cross-contamination of filters
- if filters come into contact with foreign objects and they become contaminated, they must either be flagged (if the contamination occurred during postsampling activities) or rejected from further sampling activities (if contamination occurred during presampling activities).
- once a filter is inspected and accepted, it should only come in contact with the conditioning Petrislide, forceps, antistatic strip, or weighing pan until a preweight is established
- once a preweight is established, the filter should only come in contact with forceps, the Petrislide, and the sampling cassette until it is inspected during laboratory receipt COC procedures.
- once the filter has been inspected during laboratory receipt COC procedures, it should only come in contact with the conditioning Petrislide, forceps, antistatic strips, or weighing pan

7.2 Filter Inventory and Inspection

7.2.1 New Shipment, Pre-inspection Filter Inventory

The laboratory should receive a year's supply of filters approximately in November of the previous calendar year. The shipment should contain a number of filter boxes, each box containing 50 filters. In most cases, the filter box will arrive shrink-wrapped. If not, the box may have been opened by OAQPS contractors to test a subsample of the filters. Before opening any boxes, the Region 4 and Region 10 WAMs and/or the LA should inventory the filter boxes.

NOTE: The lot number of the filter box is labeled on the shrink wrap of each filter box. Do not open a filter box until the lot numbers and the box numbers are recorded.

1. Take a Filter Inventory/Inspection Form INV-02.

2. Take all filter boxes from the shipment. Locate the lot number on the plastic shrink wrap.

3. Group the boxes by lot number.

4. For each lot number, group the filters consecutively by box number.

5. Fill in Form INV-02.

6. Keep the original and file it in AIRP/486; and provide the WAM with a copy.

7.2.2 Filter Inspection

Filters will be inspected when the LA gets ready to put individual filters out for conditioning. All filters within a filter box will be inspected at the same time. Based on the number of samples required to be sent to the field regions every 2 weeks, a filter box should be inspected every month. Inspection will occur in the conditioning environment of the laboratory weighing room.

1. Put on a laboratory coat and new pair of antistatic gloves.

2. Select the appropriate Filter Inventory/Inspection Form INV-02.

3. Select the next filter box, based on the first-in/first-out sequence of acceptable filter lots, on Form INV-02 and open it.

4. With clean filter forceps, select a filter.

5. Visually inspect the filter for the following specific filter defects:
   - Pinhole - a small hole appearing as a distinct and obvious bright point of light when examined over the light table. If light appears turn filter over, if light still appears then a pinhole is identified.
   - Separation of ring - any separation or lack of seal between the filter and the filter border reinforcing the ring.
   - Chaff or flashing- any extra material on the reinforcing polyolefin ring or on the heat seal area that would prevent an airtight seal during sampling.
   - Loose material - any extra loose material or dirt particles on the filter.
   - Discoloration - any obvious discoloration that might be evidence of contamination.
   - Filter nonuniformity - any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity or density across the face of the filter.
   - Other - a filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

6. If any of these defects are discovered on the filter, reject the filter and place it in a reject pile (not in a Petrislide).

7. If the filter is acceptable put it in a Petrislide and close the dish. It will undergo further conditioning (See SOP PEPL-6.01).

8. Repeat steps 4-6 until a filter box is completely emptied. Count the number of filters accepted from the filter box. Add this value, the date, and the LA's initials to Form INV-02.

9. Repeat steps 3-8 for another filter box.

10. At the end of the inspection process, place the rejected filters into a reject envelope. Record the information from the shrink wrap onto a label along with the LA's initials and the date, and put the label on the envelope. This label will facilitate later evaluation of the rejection's cause and the details, if necessary. If the rejection rate is greater than 15%, contact the WAM.

Filter Inventory/Inspection Form							
Lot Number	Box Number	Filter ID Number Ranges		Date Received	Date Inspected	# Accepted	Lab Analyst Initials
		From	To				
Form INV-02							

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 6.0 Filter Conditioning

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Filter Conditioning

SOP: PEPL-6.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP is applicable to the conditioning of filters, both unexposed and exposed, used in performance evaluations PEs of $PM_{2.5}$ sampling methods.

2.0 Summary of Method

Filters will be conditioned in sets that will allow the laboratory to ship out samples at the appropriate time while maintaining enough space to condition exposed filters coming back from the field. These procedures describe the process for determining the length of time for conditioning, the number of samples to condition, and the conditioning of both unexposed and exposed filters.

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

4.0 Cautions

- maintain the conditioning area in a state of good order so that samples will not be contaminated, misplaced, or misidentified
- control the conditioning environment to within the specified temperature and humidity ranges (mean temperature = 20 to 23° C with ±2° C control over 24 hours, mean RH = 30 to 40% with ± 5% RH control over 24 hours). With respect to adjacent rooms or spaces, the air should flow from the balance and conditioning area to other areas or rooms
- filter the air in the conditioning environment to reduce contamination from airborne dust.
- the room must have a HEPA-filtered air supply system on its inlet air system to minimize airborne contaminants Change the filter monthly.
- maintain the room at a slightly positive pressure
- minimize ingress to and egress from the conditioning environment
- minimize dust contamination by cleaning the weighing area daily, by installing sticky floor covering on the entrance to the conditioning environment, and by wearing clean laboratory clothing over any clothing exposed to uncontrolled environments

5.0 Interferences

Some Teflon® membrane filters were found to exhibit a weight loss of up to 150 µg over a period of up to 6 weeks after they were removed from their original shipping containers. Such weight loss can effect the accuracy of the analysis of these filters. Although it is anticipated that the filters used for the PEs will not have this problem, measure the filter weight stability of any new lot of filters that is received. Do not use any filters until their weights have stabilized.

If nitric acid vapor is present at a sampling location, it can deposit on a Teflon® filter and cause small weight gains in proportion to the amount of nitric acid present in the atmosphere (Lipfert 1994). This weight gain may not be controllable. Weight losses can occur due to thermal or chemical decomposition or evaporation of compounds like ammonium nitrate (NH_4NO_3), which releases ammonia and nitric acid as gases. Semivolatile organic compounds (SVOCs) may be part of the particulate matter on the filters; if so, they may evaporate and cause filter weight losses. Minimize or standardize these weight losses by keeping the filters cool during transport to the weighing laboratory and by conditioning and weighing the filters promptly after their receipt in the laboratory.

6.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM performance evaluation PE training is required.

7.0 Apparatus and Materials

The LA will use the following apparatus and materials to perform this procedure:

- Lot Blank Filter Stability Test Form FST-01;
- Lot Exposure Blank Filter Stability Test Form FST-02;
- Postsampling Routine Filter Stability Test Form FST-03;
- Filter Data Entry Form BAT-01;
- PEP Chain of Custody Form COC-2;
- unopened, shrink-wrapped boxes of $PM_{2.5}$ filters;
- powder free antistatic gloves
- lab coat;
- filter forceps; smooth, non-serrated
- microbalance;
- filters;
- Petrislides;
- labels for Petrislides holding blank filters
- polonium strips, in single strip mounts and gooseneck holders.

8.0 Procedures

8.1 Initial Filter Lot Stability Measurements for Lot Blanks

This procedure describes the method to determine the minimum length of time to condition filters that have been opened from their shrink wrapped boxes for initial exposure to laboratory conditions. **This procedure only needs to be accomplished once with each distinct lot of filters.**

NOTE: Do not implement this method until filters have been inventoried (See SOP PEPL-5.01)

1. Follow the filter inventory procedures given in SOP PEPL- 5.01.

2. Randomly select 3 filter boxes from the inventory of filter boxes.

3. From each filter box, randomly select 3 filters. These filters will be identified as **lot blanks**. This will provide for a total of 9 filters.

4. Inspect these 9 filters as described in SOP PEPL-5.01.

5. If the filters are acceptable, place each filter in a Petrislide and identify each filter by a number on the Petrislide.

6. Take a Filter Lot Stability Test Form FST-01 and record each filter.

7. Allow these nine filters to condition for 24 hours

8. After 24 hours, weigh the filters following SOP PEPL-8.01 every 24 hours for a minimum of 5 days **and** until the difference between one weight and the consecutive weight of all 9 filters is less than 15 μg. If the test goes longer than 5 days, take another Filter Lot Stability Test Form FST-01 and continue to record the data.

9. Record the length of time (in days) it took each filter to stabilize.

10. Calculate the average length of time for the 9 filters to stabilize. This average will provide the LA with an estimate of the time it will take to bring filters in a box that have just been opened to a constant weight.

    Note: It is important to note any trends during this test. Control charting this data can help. Upon weighing, if decreasing weights continuously occur over the 5 day period and longer, even though it may be less than 15 ug, this will indicate outgasing which is why conditioning

is important. The LA may want to continue this test until this downward trend stops or is negligible (5 ug difference). If the trend is an increase in weight, it may indicate laboratory contamination, which must be rectified.

8.2 Individual Filter Conditioning - Presample weighing

Section 8.1 established the typical length of time that a box of filters, once opened, should be set out into the conditioning environment. This procedure describes the method for determining that a set of filters that will be used for a presampling weighing session have been sufficiently conditioned (stable) that the LA can proceed with weighing them for shipment to the field.

1. From the Filter Inventory/Inspection Form INV-02 select the next set of filter boxes for individual filter conditioning.

2. Inspect the filters as described in SOP PEPL-5.01.

3. Place the acceptable filters in individual Petrislides and place the dishes in the conditioning environment. Place the lids 3/4 over the filters.

4. Group these filters in an area where the LA can identify when these filters were set out.

5. Allow the filters to condition for the length of time established in Section 8.1 above.

6. From the set of filters that have been placed out at one time, select three filters at random. These filters will be identified as **lot exposure blanks**.

7. Select a Lot Exposure Blank Filter Stability Form FST-02.

8. Weigh the lot exposure blank filters and QC check weights following SOP PEPL-8.01 and record the initial weight.

9. Maintain the required temperature and RH. Measure temperature and RH on a continuous basis during filter weight stability experimentation using electronic instruments whose outputs are recorded by a data acquisition system connected to a computer. Calculate the means and ranges of the conditioning environment's temperature and RH over each 24-hour period, and record these values in the Laboratory Information Management System (LIMS) and/or in the laboratory QC notebook.

10. Wait 24 hours and then weigh the three lot exposure blanks again. Record the difference in micrograms from the consecutive weights.

11. Weigh the lot exposure blanks every 24 hours until the average difference between all three weights is 5 μg and no filter difference is greater than 15 μg. Once this occurs, the filters associated with the lot exposure blanks are ready for weighing.

8.3 Batch Development and Postsample Conditioning

8.3.1 Filter Batching

This procedure describes the method for batching exposed filters. The LA will develop a batch of filters that will undergo post sample conditioning and weighing and will be composed of 15 filters which would include;

- ~10 routine filters,
- at least one field blank (See also Table 9-1, in SOP PEPL 9.01, for biweekly number of sampling and blank filters needed for preparation and shipment to the field) ,
- at least one laboratory (equals lot exposure) blank ,
- a filter from the previous batch (PD)
- QC samples (working mass QC samples, duplicates etc.)
- a collocated sample filter (optional)

Note: Region 4 is estimated to process 69 (sample and blank) filters per month and Region 10 is estimated to process 58. See PEPL-4.01 Table 4-4..

1. Review the COC forms.

2. Select filters for a batch, starting with the filters that were received on the earliest date.

3. Select the lab blanks that went through the presample weighing process (PEPL-8.01) with the routine filters in this batch. There may be multiple lab blanks associated with the batch.

4. Fill in Filter Batch Data Entry Form BAT-01.

8.3.2 Filter Batch Postsample Conditioning

This procedure describes the method for conditioning a batch of exposed filters that have returned from the field. Filters may or may not have been stored in a refrigerator.

1. Allow filters from the refrigerator to equilibrate to room temperature for 12 to 24 hours in the laboratory

NOTE: Because these filters and the containers are probably colder or warmer and/or significantly more or less humid than the conditioning area or room, do **not** place them, immediately upon receipt, in the conditioning environment where they might affect the temperature or humidity requirements of filters being conditioned. Because the filters are still in their plastic shipping bags, they will not be contaminated.

2. Follow the filter receiving COC procedures described in SOP PEPL-10.01.

3. Condition exposed filters in the Petrislide for at least 24 hours under the same environmental conditions (i.e., mean RH within ±5 %) as existed during presampling conditioning.

4. After 24 hours, select 3 routine filters.

5. Select a Postsampling Routine Filter Stability Test Form FST-03.

6. Weigh the 3 routine filters and QC check weights following SOP PEPL-8.01, and record the initial weight.

7. Maintain the required temperature and RH. Measure temperature and RH on a continuous basis during filter weight stability experimentation using electronic instruments whose outputs are recorded by a data acquisition system connected to a computer. Calculate the means and ranges of the conditioning environment's temperature and RH over each 24-hour period, and record these values in the LIMS and/or in the laboratory QC notebook.

8. Wait a minimum of 8 hours and then weigh the routine samples again. Record the difference in micrograms from the consecutive weights.

9. Weigh the 3 routine sample filters every 12-24 hours until the difference between two out of the three filters consecutive weights is less than 15 μg. Once this occurs, the batch associated with the 3 filters is ready for weighing.

10. Follow the postsampling filter weighing procedures given in SOP PEPL-8.01.

PEP Lot Blank Filter Stability Test

Lot # Represented _____ Filter Box Numbers Represented _____ Lab Analyst Initials _____

		Date		Date		Date		Date		Date	Total Days	Average
			1-2		2-3							
	Filter ID	Day1 mg xxx.xxx	Diff μg xxx	Day 2 mg xxx.xxx	Diff μg xxx	Day 3 mg xxx.xxx	Diff μg xxx	Day 4 mg xxx.xxx	Diff μg xxx	Day 5 mg xxx.xxx		
QC1 100 mg												
QC2 200 mg												
Filter 1												
Filter 2												
Filter 3												
Filter 4												
Filter 5												
Filter 6												
Filter 7												
Filter 8												
Filter 9												
QC1 100 mg												
QC2 200 mg												
FST-01												

PEP Lot Exposure Blank Filter Stability Test

Filter Box Numbers Represented _____ Lab Analyst Initials _____

		Date		Date		Date		Date		Date
			1-2		2-3					
	Filter ID	Initial weight mg xxx.xxx	Diff μg xxx	Day 1 24 hours mg xxx.xxx	Diff μg xxx	Day 2 48 hours mg xxx.xxx	Diff μg xxx	Day 3 72 hours mg xxx.xxx	Diff μg xxx	Day 4 96 hours mg xxx.xxx
QC1 100mg										
QC2 200mg										
Filter 1										
Filter 2										
Filter 3										
QC1 100mg										
QC2 200mg										

FST-02

PEP Postsampling Routine Filter Stability Test

Filter Batch Number Represented _____ Lab Analyst Initials _____

		Date		Date		Date		Date		Date
			1-2		2-3					
	Filter ID	Day 1 24 hr weight mg xxx.xxx	Diff μg xxx	Day 2 12-24 hours mg xxx.xxx	Diff μg xxx	Day 3 12-24 hours mg xxx.xxx	Diff μg xxx	Day 4 12-24 hours mg xxx.xxx	Diff μg xxx	Day 5 12-24 hours mg xxx.xxx
QC1 100 mg										
QC2 200 mg										
Filter 1										
Filter 2										
Filter 3										
QC1 100 mg										
QC2 200 mg										

FST-03

PEP Filter Weighing Data Entry Form

Batch Type: PRE POST Batch No. _____

Date _____ Analyst Initials _____

Mean Temp for Past 24 hours: _____ SD:_____
Mean RH for Past 24 hours: _____ SD:_____

Sample	Filter ID	Filter Type 00/ LB/FB CO/BD/PD	Cassette ID	Weight 1 xxx.xxx mg	Weight 2 xxx.xxx mg	Flag
QC1	100 mg					
QC2	200 mg					
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Filter						
Duplicate 1		BD				
Duplicate 2		DU				
Duplicate 3		DU				
QC1	100 mg					
QC2	200 mg					
BAT-01						

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 7.0 Calibrations

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Quarterly Verification of Working Mass Reference Standards

SOP: PEPL-7.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP will be used every 3 months to compare the working standards against the laboratory primary standards to ensure that the working standards are still within the acceptance criteria of their certified weight. This method is applicable to all weighings using a single-pan, fully electronic Sartorius Model MC5 microbalance that combines digital indications with the use of a built-in (internal) weight (5 g)**,** and contains software for autozero, internal calibration, and auto correction for air bouyancy and temperature sensitivity adjustment, if needed.

2.0 Summary of Method

The double-substitution procedure is one in which a standard (primary standard) and an unknown weight (working standard) are intercompared twice to determine the average difference between the two weights. Errors in any built-in weights or in the balance indications are eliminated by using the balance only as a comparator and by calibrating the balance indications over the range of use for the measurement. Accordingly, the procedure is especially useful for high-accuracy calibrations. The procedure is a version of SOP No.4 in NIST Handbook No.145 tailored to this specific application and equipment configuration.

The working and primary standard weights are each weighed twice. First, the working standard is weighed, and then the primary standard is weighed. Then the primary weighing is repeated; and finally the working standard is weighed again. All weighings are made at regularly spaced time intervals to average out any effects of instrument drift.

3.0 Definitions

The following symbols are used in this procedure:

p = the PE lab "primary" standard weight.
w = the working standard weight to be verified.
M = the mass of a specific weight. Subscripts p, and w are used to identify the weight.
AM = the apparent mass of a specific weight. Subscripts p, and w, are used to identify the weight**.**

4.0 Interferences

Due to the weights of the standards being small, contamination of the weights and/or microbalance can affect the success of the calibration. It is recommended that, immediately before performing this procedure, the laboratory analyst (LA) completely clean or cover body areas that might be sources of contamination

The LA must wear a clean lab coat over potentially contaminated clothes and must wear disposable, non-contaminating, powderless antistatic gloves. No one who is coughing or sneezing should be allowed in the weighing/conditioning room.

5.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training.

6.0 Apparatus and Materials

ASTM Class 1 mass reference standards must be used for verifying the working standard weights. Mass standards must be available with calibration certificates traceable to NIST. The balance must be in good operating condition as verified by a valid control chart or preliminary experiments to ascertain its performance quality. Keep the working standard weights in their box by the Sartorius Model MC5. Store the laboratory primary standard weights in an air-tight, air-resistant (or at least fine particulate-excluding), limited access or locked compartment.

The following equipment is required to perform this procedure:

- Sartorious MC5 microbalance
- ASTM Class 1 NIST-traceable working standard weight set (including 100 mg and 200 mg)
- ASTM Class 1 NIST-traceable lab primary standard weights (100 and 200 mg)
- mass forceps, non-metallic (one for each set)
- a stop watch or other timing device to observe the time of each measurement
- powder-free laboratory antistatic gloves
- laboratory coat
- Quarterly Standards Verification Form (Form QSV-01)
- Alcohol swabs

7.0 Instrument Verification

7.1 Preliminary Procedure

1. Wipe down the work area with an alcohol swab.

2. Place the laboratory primary standard weights next to the working standard weights near the balance overnight to permit the weights and the balance to attain thermal equilibrium.

3. Conduct preliminary measurements with the 200-mg working-standard weight to warm up the microbalance by weighing with a typical load -this is called exercising- and to verify that the time interval required for the balance indication to stabilize is still on the order of 20 seconds (much less than a minute). Repeat this procedure a few times.

7.2 Verification Procedure

7. Zero using the TARE/ key and calibrate using the F1/ key according to the Sartorious *Installation and Operating Instructions* (see pages 1-22 and 1-28).

8. Open the draft shield

9. Using the cleaned, labeled standard non-metallic weight forceps, gently place the 200 mg working standard (w) on the sample pan.

10. Close the draft shield. Wait until the display of the selected unit of weight indicates that a stable reading has been obtained. Time twenty seconds and if the weight remains stable, record the weight on the Quarterly Standards Verification Form as measurement 1

11. Open the draft shield and remove the weight using the non-metallic weight forceps.

12. Shut the draft shield and allow the microbalance to come to zero. Wait at least 20 seconds to ensure zero is achieved. If not achieved by then, the instrument can be manually zeroed using the TARE key.

13. Repeat steps 2-6 for the 200-mg primary (p) and working standard (w) weights in order to weigh each standard 2 times. Note that the primary standard is weighed consecutively.

Measurement No.	Weights on Pan	Observation No.
1	*w*	O_1
2	*p*	O_2
3	*p*	O_3
4	*w*	O_4

NOTE: The time intervals between successive trials should not differ from one another by more than ±20%. If this difference is exceeded, reject the data and take a new series of measurements that so agree.

14. Repeat steps 2-6 for the 100 mg weight

15. Calculate C_w (see subsection 9.1)

16. Subsequent measurements of C_w must be with $\pm$ 2 ug of the initial C_w value

8.0 Troubleshooting

If the test results do not indicate acceptable agreement (2 ug) and a repetition produces the same results, use SOP PEPL 7.02 to confirm that the external primary standard (5g) and the Model MC5's internal standard (5g) still agree. If they do, arrange to have the primary standard weight checked against an independent, certified weight of the same or greater confidence level of accuracy. If they don't agree, the WAM should authorize consultation with the service technician.

9.0 Data Acquisition, Validation, Calculations, and Data Reduction

9.1 Calculations

Calculate the apparent mass correction, C_w, for the test (working standard) weight (*w*) as follows, according to the sequence used. In each case, the apparent mass corrections for the primary standard weight, C_p , are included. The symbols N_p and N_w refer to the nominal values of *p* and *w*, respectively.

$$\mathbf{C_w = C_p + ((O_1 - O_2 + O_4 - O_3)/2) + N_p - N_w} \quad \text{Equation 1}$$

9.2 Assignment of Uncertainty (Optional)

The limits of uncertainty, U, include estimates of the mass standards used, U_s , plus the uncertainty of the measurement, U_m , at the 99.73% level of confidence. U_m is estimated by

ts Equation 2

where *s* is the standard deviation of measurement and *t* is obtained from Table 9.3 (see attached).

Then

$$U = \pm [\, U_s + ts \,] \quad \text{Equation 3}$$

9.2.1 Precision of Measurement Known from Control Chart Performance (Optional) (See SOP No. 9 in NIST Handbook No. 145**.)**

The value for *s* is obtained from the control chart data. Statistical control will need to be verified by measuring at least one check standard when the above measurements are being taken.

Use the value of *t* (corresponding to a probability level of 99.73%) from Table 9.3 (NIST Reference) appropriate for the number of degrees of freedom *(df),* ν , on which the control limits of the control chart are based. For each measurement on a control chart, identify the number of measurements used to obtain that value (*n*) and subtract 1 (df = ν = *n-1*).

9.2.2 Precision Estimated from Series of Measurements (Optional)

Measure a stable test object at least seven 7 times, no two measurements of which may be on a single day. Calculate the mean and the standard deviation in the conventional manner, which is already provided in the text and equations of SOP PEPL 2.01, subsection 10.2. The equations are the same as those in NIST Handbook No. 145, where they are used to calculate the value of *s* that is used in section 4.4 of SOP No. 9 in Handbook No. 145. In this case select the value for *t* from table 9.3 based on the number of degrees of freedom involved in computing *s*.

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation.

Quarterly Standard Verification Form

Test No. : _____ Form(at) No. : _____ , Date :_____

Item Identification:_____ Balance :_____

Standard Identification:_____Observer:_____

c_p (200 mg)= _____ ± _____ c_p (100 mg)= _____ ± _____

Time :_____ Balance Standard Deviation : _____

200 mg Test

Measurement No.	Weights	Observations
1	*w*	O_1 =
2	*p*	O_2 =
3	*p*	O_3 =
4	*w*	O_4 =

100 mg Test

Measurement No.	Weights	Observations
1	*w*	O_1 =
2	*p*	O_2 =
3	*p*	O_3 =
4	*w*	O_4 =

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: External Calibration of Analytical Microbalance

SOP: PEPL-7.02

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP describes an external calibration of a Sartorius Model MC5 microbalance. **An external calibration should be performed ONLY when routine quality control (QC) checks (e.g., microbalance verifications using working standards, replicate filter weighings) indicate that the microbalance may be out of calibration and is approved by the WAM.**

2.0 Summary of Method

The microbalance's software is set so that weighing a 5-g primary mass reference standard with NIST traceability will produce a digital display of 5 g.

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP. For this SOP, "external calibration" is a one-point calibration of the microbalance using a mass reference standard.

4.0 Cautions

To ensure maximum stability, leave the microbalance turned on at all times. This procedure will enable the microbalance to be operational at any time and will eliminate the need for a warmup period before analyses can be performed.

Have the microbalance calibrated on a regular basis (e.g., twice yearly) by a microbalance service technician and maintain it according to the manufacturer's recommendations. The calibration should be traceable to NIST-traceable mass reference standards.

If the microbalance cannot be calibrated according to this SOP, have it adjusted and repaired by an authorized microbalance service technician according to the manufacturer's directions. Do not attempt to adjust or repair the microbalance.

Use smooth, nonmetallic forceps for handling mass reference standards. Handle the standards only with these forceps and do not use the forceps for any other purpose. Mark the forceps to distinguish them from the forceps used to handle filters. Clean the forceps with alcohol and lint-free wipes and then allow them to air-dry before handling the standards. Handle the standards carefully to avoid altering their masses by damage or contamination.

This procedure uses the primary standards, not the working standards.

5.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training is required.

6.0 Apparatus and Materials

The following equipment are required to perform this procedure:

- analytical microbalance
- NIST-traceable, laboratory **primary** mass reference standard (ASTM Class 1)
- mass non-metallic forceps for handling the standard
- Powder-free laboratory antistatic gloves
- laboratory coat

7.0 Procedure

7.1 Instrument or Method Calibration

1. Turn off the balance display using the ON/OFF key and then turn it back on. While all segments are displayed, briefly press the PRINT key. The balance model should be displayed. Press the CF key to display the serial number. Record the balance model and serial number in the laboratory QC notebook. Press the CF key again to quit this function.

2. Unload the weighing pan and close the draft shield.

3. Press the TARE key for at least 2 seconds until "C.I." and "CAL" display.

4. Press the F2 key until "C.E." (for external calibration) displays.

5. Press the TARE key again. When a zero readout is displayed, press the F1 key. The calibration weight readout now will be in grams.

6. If external interference affects the calibration procedure, the error message "Err02" will appear briefly. In this case, repeat step 5.

7. Enter the certified mass of the mass reference standard using the numeric keys. Then press the F1 key identified on the weight display by "STO" to store this mass value. Enter the date and time, the certified mass of the mass reference standard, and the standard's identification number in the laboratory QC notebook.

8. Open the draft shield and transfer the standard to the center of the weighing pan using the forceps. Close the draft shield. The microbalance will generate an audible signal when it has obtained a stable measurement of the standard's weight.

9. Open the draft shield and transfer the standard back to its protective container. Close the draft shield.

8.0 Troubleshooting

Consult the *Installation and Operating Instructions* for the microbalance. The document's troubleshooting guide is on pages 1-33 and 1-34. To assess affects of temperature on weighing sensitivity, use the calibration test on pages 1-37and 1-38. If the balance may have been moved, use the test on page 1-39.

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Quarterly Verification of Recording Thermometer and Recording Hygrometer

SOP: PEPL-7.03

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

The recording thermometer and the recording hygrometer of the environmental control system for the $PM_{2.5}$ conditioning and weighing room measure and display the temperature and RH inside the $PM_{2.5}$ conditioning environment on a continuous basis. These instruments' responses are recorded continuously by a computer-based data acquisition system. The accuracy of the responses is verified by comparison testing at least one % RH value -the set point, usually 35% RH- on a quarterly basis using a laboratory reference standard, the instant model of the Fisherbrand™ Certified Traceable Digital Hygrometer/Thermometer (DH/T).

2.0 Summary of Method

The laboratory reference, instant model Fisherbrand™ Certified Traceable Digital Hygrometer/Thermometer (DH/T), is placed inside the conditioning environment, which is allowed to vary, during a 24 hour period, up to a±2 °C control limit within an allowed 20 to 23°C operating range and up to ±5% RH control limit within an allowed 30 to 40% RH operating range. The responses of the reference instrument's combination probe are then compared with the responses of the conditioning environment control system's recording thermometer and recording hygrometer. Mean and standard deviation are calculated from the recorded responses. The mean is compared to the operating range and must be within it. The standard deviations are converted to the appropriate T or RH units and compared to the control limits and must be within them.

The laboratory reference DH/T is a combination probe containing two sensors. One sensor is a thermistor that is calibrated by the manufacturer against a NIST traceable temperature so that it measures the ambient or [”dry-bulb”] temperature in the weighing and conditioning room. The digital hygrometer component of the probe is a precision, thin-film (semipermeable membrane) capacitance sensor. The circuitry of the combination probe uses the precalibrated thermistor output and the calibrated capacitance change resulting from the amount of moisture allowed into the sensor by the semipermeable membrane to calculate the RH. The digital thermometer and RH probe components measure and the LCD and computer readouts display the selected high, low or current values.

This procedure also verifies that the conditioning environment‘s temperature and humidity control systems are operating within their control limits.

3. 0 Verification Acceptance Criteria

Table 8-2 of the Implementation Plan (8/98) for the $PM_{2.5}$ FRM PEP presents the following acceptance criteria for laboratory QC checks:

Temperature calibration	±2°C
Humidity calibration	±2% RH

The certified accuracies of the Fisher brand ™ instant model of the DH/T are ± 0.2°C and ± 1.5 % RH which, in this application, means that the Fisherbrand instant model of the DH/T meets the general standard verification requirement that a verification standard must be as accurate or more accurate than the acceptance criteria it is being used to verify. The certification of these accuracies are important. Therefore the certificate that comes with the DH/T to provides this quality assurance should be checked when it arrives by confirming with NIST that any reference to NIST testing is correct.

4.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

For this SOP, calibration is distinguished from verification as follows: Calibration involves a multi-point characterization of a sensor's response and adjustment, if necessary, to bring its output into agreement with a NIST-traceable standard throughout the calibration range. Verification typically involves a single or multipoint check of a sensor's response to verify that it is functioning within a tolerance range.

5.0 Cautions

RH values inside the conditioning environment will change as the temperature varies, as shown in the Background Information subsection located at the end of this section. Within the operating range of the conditioning environment, a 1°C change in the temperature will produce an approximately 5% RH change. Remain aware of this interaction of verification parameters as temperature and humidity levels inside the conditioning environment are varied.

The LA should be sure to utilize the arrangement to have the certified calibration of the laboratory reference DH/T verified annually using NIST-traceable temperature and/or humidity reference standards by the manufacturer or at a State weights and measures laboratory holding a NIST Certificate of Traceability or at a calibration laboratory that is accredited by the National Voluntary Laboratory Accreditation Program (NVLAP).

The humidity sensor, a precision thin film (semipermeable membrane) capacitance-based sensor, and the temperature sensor, a thermistor, are extremely unlikely to be operated, even accidentally,

outside of their very wide operating range. However since the combination probe and LCD readout are powered by a 9V alkaline battery with a 1 year intermittent and 100 hour continuous use limitation, attention should be given to the low battery indicator as the use time of the system approaches the specified use limits. Since the system is based on electronic circuitry, check for effects of any damage to the protective outer housings of the probe, cable, or LCD readout components.

6.0 Personnel Qualifications

Certification of passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training is required.

7.0 Apparatus and Materials

The following equipment is required to perform this procedure:

- laboratory reference Fisherbrand™ instant model DH/T
- humidifiers and/or dehumidifiers, if the conditioning environment does not have a humidification capability
- provision for manual or computer data recording.

8.0 Instrument Verification Procedure

NOTE: Be sure to read the environmental control system and DH/T manufacturer's operation instructions before starting this procedure.

1. Install the laboratory reference DH/T in the conditioning environment. Locate the combination probe as close as possible to the temperature and RH sensing components of the environmental control system of the Laboratory's conditioning and weighing area. In considering sensor placement based on the air flow patterns in the controlled area, place the reference standard probe downstream of the control system's sensors and upstream of the microbalance, so that the air entering the controlled area goes first by the reference probe and then, without obstruction, by the balance.

2. Turn on the DH/T by pressing the ON key on the face of the LCD /control component of the system and allow this instrument to stabilize. When the unit is initially turned on, it will be in the Auto Scroll mode. It will display a reading, pause, and then scroll to the next measurement. Press the Hold part of the Hold /Scroll key to stop the scrolling. The display will only read the present measurement.

3. When the recording thermometer and recording hygrometer readings indicate that the temperature and RH have stabilized, record, manually or by computer, the readings of the

recording thermometer, the recording hygrometer, and the DH/T's T and RH probe (on the LCD readout screen or receiving monitor of the DH/T computer output. Record in the laboratory QC notebook (paper or electronic form), in data format shown in Figure 7.03-1 below.

4. Calculate the difference between the recording thermometer measurement and the reference instrument measurement for the conditioning environment setting and compare this difference to the temperature verification acceptance criterion of ±2°C.

5. Calculate the difference between the recording hygrometer measurement and the laboratory reference DH/T measurement for the conditioning environment settings and compare this difference to the T and RH verification acceptance criterion of ±2% RH.

Figure 7.03-1: T, RH 1-pt Monthly Verification Data Form

Date_____ LA or Svc. Technician_____ T-STD Model No._____

T-STD Ser. No._____ RH STD Model No. _____ RH-STD Ser.No._____

Recording T/RH Model No._____, Ser.No._____
Record of Test Observations(5 min. Avg., at least 1 hr.):

Mean Recording T (°C) =_____, S.D. =_____ ; Mean Std. T (°C) =_____, S.D.=_____
Mean Diff. (°C)=_____

Mean Recording %RH =_____, S.D.=_____ ; Mean Std % RH =_____, S.D.=_____
Mean % Diff =_____

The T and RH may vary outside of the allowed mean ranges of 20 to 23 °C temperature or 30 to 40 % RH or the 24 hour allowed variability ranges of ±2°C or ±2% RH during the year for periods longer than 5-10 minutes during changes, such as door openings, multiple person entry or presence, etc. This may occur more commonly in some geographical and seasonal situation s than others, such as high heat and humidity in Athens, GA in August. If this occurs, allow the DH/T to measure, and the LA should record the high, low, and current values, so that mean and standard deviation for more than one point can be computed and compared as above to the output of the environmental control system's output for the same period.

9.0 Troubleshooting

1. If the measurements of the recording thermometer and the reference instrument disagree by more than the temperature verification acceptance criterion at the conditioning environment setting or if stable readings cannot be obtained, reverify the recording

thermometer measurement. If the two readings still disagree, investigate the recording thermometer and the reference instrument and take appropriate corrective action. This corrective action may include the having the LA ask the WAM to authorize previously arranged facility or external service technician support in (1) adjusting the calibration of the recording thermometer to agree with the reference instrument, (2) repairing the recording thermometer, and/or (3) recertifying the recording thermometer and reference instrument against a NIST-traceable temperature standard at a State weights and measures laboratory holding a NIST Certificate of Traceability or at a calibration laboratory accredited by NVLAP.

If the recording thermometer cannot be calibrated, make note of this problem in the laboratory QC notebook. Attach a copy of the service technician's report.

2. If the measurements of the recording hygrometer and the laboratory reference DH/T hygrometer disagree by more than the RH verification acceptance criterion at the conditioning environment setting or if stable readings cannot be obtained, reverify the hygrometer measurement. If the two readings still disagree, investigate the hygrometer and the laboratory reference psychrometer and take appropriate corrective action. This corrective action may include having the LA ask the WAM to authorize previously arranged facility or external service technician support in (1) adjusting the calibration of the recording hygrometer to agree with the laboratory reference psychrometer, (2) repairing the hygrometer, and/or (3) recertifying the laboratory reference psychrometer using NIST-traceable temperature or humidity reference standards at a State weights and measures laboratory holding a NIST Certificate of Traceability or at a calibration laboratory accredited by NVLAP.

   If the recording hygrometer cannot be calibrated, make note of this problem in the laboratory QC notebook. Attach a copy of the service technician's report.

3. If the conditioning environment cannot maintain the temperature and RH at the specified setting, troubleshoot the control systems in the conditioning environment and take appropriate corrective action. This corrective action may include having the conditioning environment repaired by an authorized service representative from the company that manufactured the conditioning environment system. If the conditioning environment cannot be controlled , make note of this problem in the laboratory QC notebook.

Background Information

If questions arise regarding the T or RH readings of electronically-based sensors, NIST recommends comparison for T to a recently certified, NIST-traceable (or recertified, NIST traceable) dry bulb thermometer; or for RH to a series of salt solutions (according to ASTM standard procedure E104. **The LA is not expected to perform such tests.**

Service agreements and annual recertification agreements have been arranged by the WAM in case problems or questions arise that suggest the need for such tests. This information and the information provided below are just included here as background knowledge.

The relationship between the dry-bulb temperature, the wet-bulb temperature, and the RH in the conditioning environment's operating range is given in the following table. Dry-bulb temperatures appear across the top of the table, wet-bulb temperatures appear down the left side of the table, and RH values appear in the interior cells.

Wet-Bulb Temperature (°C)	Dry-Bulb Temperature (°C)						
	20.0	20.5	21.0	21.5	22.0	22.5	23.0
10.7	29.2	26.9	24.8	22.8	20.9	19.1	17
11.2	32.4	30.1	27.9	25.7	23.7	21.8	20.0
11.7	35.7	33.3	31.0	28.8	26.7	24.7	22.8
12.2	39.1	36.5	34.1	31.8	29.6	27.6	25.6
12.7	42.5	39.8	37.3	34.9	32.7	30.5	28.4
13.2	46.0	43.2	40.6	38.1	35.7	33.5	31.3
13.7	49.5	46.7	43.9	41.3	38.9	36.5	34.3
14.2	53.1	50.1	47.3	44.6	42.0	39.6	37.3
14.7	56.8	53.7	50.8	48.0	45.3	42.7	40.3

If you know the dry-bulb temperature (T_{db}) and the wet-bulb temperature (T_{wb}), and want to obtain the RH (in percent), use these formulas as follows:

$$RH = 100\ [\rho_{ws}(T_{wb})/\rho_{ws}(T_{db})] \qquad (7\text{-}4)$$

$$\rho_{ws}(T_{wb}) = \exp[(C_1/T_{wb}) + C_2 + C_3T_{wb} + C_4T_{wb}^{\ 2} + C_5T_{wb}^{\ 3} + C_6\ln(T_{wb})] \qquad (7\text{-}5)$$

= water saturation vapor pressure (in psia) at the wet-bulb absolute temperature (in Kelvin, ° R = ° F + 459.67)

$$\rho_{ws}(T_{db}) = \exp[(C_1/T_{db}) + C_2 + C_3T_{db} + C_4T_{db}^{\ 2} + C_5T_{db}^{\ 3} + C_6\ln(T_{db})] \qquad (7\text{-}6)$$

= water saturation vapor pressure (in psia) at the dry-bulb absolute temperature (in Kelvin, ° R = ° F + 459.67)

where

$C1 = -1.0440397 \times 10^{+4}$,
$C2 = -1.1294650 \times 10^{+1}$,

$C3 = -2.7022355 \times 10^{-2}$,
$C4 = +1.2890360 \times 10^{-5}$,
$C5 = -2.4780681 \times 10^{-9}$, and
$C6 = +6.5459673 \times 10^{+0}$.

Woods Hole Oceanographic Institution has measured RH at the sea surface on buoys using thin-film polymer, capacitative sensors. Between 1990 and 1994, 14 sensors were calibrated on a regular basis using an RH chamber with a calibration accuracy of approximately 0.3% RH at 40% RH. During an approximately 600-day period since their calibration, the sensors' responses were verified at nominal RHs ranging from 20 to 90% RH at 10% RH intervals. During this period, 35 independent verifications were obtained at 40% RH. The sensors had random shifts in their calibrations of ±2 to 3% RH at 40% RH. There did not appear to be any long-term drifts.

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 8.0 Filter Weighing

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Filter Weighing

SOP: PEPL-8.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP applies to the presampling and postsampling weighing of filters used in the $PM_{2.5}$ PEP. It also provides information for preparing the filters for shipment to the field.

2.0 Summary of Method

Filters are conditioned and once they have reached equilibrium are batched (PEPL-6.01) and weighed either to record a presampling weight or a postsampling weight. Various types of QC samples are also weighed and reviewed in order to accept the data from the weighing session.

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

4.0 Cautions

- filters should be weighed as soon as there are enough samples received to make a batch but must be weighed within 10 days of sample receipt
- keep the microbalance in the same controlled-environment room that will be used to condition the filters
- ensure the laboratory has a HEPA-filtered air supply system on its inlet air system to minimize airborne contaminants
- change the filter monthly
- maintain the room at a slightly positive pressure so that the air flows away from the balance and conditioning area
- minimize ingress to and egress from the room
- minimize dust contamination by cleaning the weighing area daily; by installing, using, and as needed, replacing sticky floor coverings (minimum weekly) at the entrance to the room; and by wearing clean laboratory clothing over any clothing exposed to uncontrolled environments
- place the microbalance on a balance table to reduce vibrations
- avoid bumping the microbalance to prevent disturbing its calibration
- verify that the analytical microbalance has been calibrated by an authorized microbalance service representative within the past 6 months
- if more than one microbalance is used, make the pre- and postsampling measurements of each filter on the same microbalance
- verify that the working standards have been either recertified within the past year against NIST-traceable mass standards at a State weights and measures laboratory holding a NIST Certificate of Traceability or at a calibration laboratory accredited by NVLAP and verified within the past 3 months against the laboratory primary standards at the weighing laboratory according to SOP PEPL-7.01

- check the working standards' masses, after any incident of rough handling, and compare to their verified value.
- select working standards so that they will bracket the mass of a blank or loaded filter (that is, 100 and 200 mg)
- when weighing filters, hold the filter (with filter forceps) only by the outer polyolefin supporting ring, not by the filter material
- if the forceps ever touch the filter material of an exposed filter, flag the sample with the code "LAC" on PEP Filter Data Entry Form BAT-01 and clean the forceps with disposable laboratory wipes to avoid cross-contamination

5.0 Interferences

Temperature effects the performance of the microbalance. Relative humidity effects moisture content and therefore the weight of the filter.

An important limitation of the method involves changes in the weight of a collected sample due to mishandling, chemical reactions, and volatilization. Handling procedures, choice of filter media, humidity and temperature control of the filter and sample during weighing, and promptness in weighing the sample following collection, all help control filter artifacts.

The chemical makeup of the $PM_{2.5}$ particulate matter varies with the sampling location and source. Thus, the magnitude of $PM_{2.5}$ weight changes due to chemical and physical processes will also vary with site location.

Minimize weight loss due to mechanical removal of particles from the filter by removing the filter from its cassette carefully, by conditioning the filter, and by neutralizing the electrostatic charge buildup on the filter before weighing.

Filters may become contaminated during conditioning or weighing from particulate matter in the air or from dust on the microbalance or working surfaces. Reduce airborne contamination by passing the air inside the conditioning environment through a HEPA filter. Reduce surface contamination by cleaning work surfaces with low-lint, disposable laboratory wipes before weighing the filters.

Stable readings of weights are a requirement. Consult the microbalance's operating manual for more information about obtaining stable readings.

5.1 Electrostatic Charge Neutralization

Errors in the gravimetric analysis of samples can also result from the buildup of an electrostatic charge on the microbalance or on filters during their manufacture or during sampling (Engelbrecht et al. 1980). This electrostatic charge buildup will interfere with the microbalance weighing. Reduce the electrostatic charge on the microbalance by electrically grounding the microbalance

and coating the nonconductive surfaces with an antistatic solution. Reduce the electrostatic charge on filters using polonium-210 (^{210}Po) antistatic strips immediately before the weighing process begins.

Common symptoms of a problem with electrostatic charge include **noisy readout, drift**, and **sudden readout shifts**. To reduce static charge on individual filters and within the microbalance, place a radioactive antistatic strip containing a very small amount (i.e., 500 picocuries) of ^{210}Po in the weighing chamber. ^{210}Po antistatic strips neutralize electrostatic charges on items brought within an inch of them. These antistatic strips are safe, commonly available, and inexpensive. ^{210}Po has a half-life of 238 days. Change the antistatic strips every 6 months and discard the old strips according to the manufacturer's recommendations and State and local regulations, if applicable.

Use antistatic solutions for coating (and, at appropriate and relatively infrequent intervals, recoating) the interior and exterior nonmetallic surfaces of the weighing chamber. This coating facilitates the draining of electrostatic charges away from these surfaces (by making them conductive) to a common ground to which the metallic conductive surfaces are connected. Place earth-grounded conductive mats on the balance table surface and beneath the analyst's shoe surfaces to reduce electrostatic charge buildup.

Even though a filter weight might stabilize within 30 to 60 seconds and no weight drift is observed during that period, the microbalance may still be influenced by some electrostatic buildup. It may still be necessary to repeat the neutralization procedure. Charge neutralization times may need to be longer than 60 seconds for sampling situations when either a high amount of charge has developed on collected particles due to their origin or the particle loading on a filter is large. Electrostatic charge buildup increases as the air becomes drier. A 60-second charge neutralization may be sufficient in ambient indoor air conditioned to 37% RH and 23° C but not in 20% RH and 23 °C in arid environments. The latter environment may require that the filter sit for more time near the antistatic strip.

6.0 Personnel Qualifications

Certification by having passed the written examination and the hands-on practical examination for the laboratory component of the $PM_{2.5}$ FRM PE training are required.

7.0 Apparatus and Materials

The following equipment is required to perform this procedure:

- Filter Weighing Form BAT-01
- Filter Inventory and Tracking Form COC-01
- filters
- microbalance

- high purity ethanol
- QC mass working standards
- working standard forceps
- filter forceps
- filter cassettes
- metal filter caps
- plastic shipping bags
- sample labels
- premoistened wipes or lint-free cloths and a small bottle of ethanol
- polonium strips
- powder free antistatic gloves
- laboratory coats

8.0 Procedures

8.1 Prerequisites

8.1.1 Filter Weighing Readiness

Follow the presampling filter handling procedures in SOP PEPL-5.01 and the presampling filter conditioning procedures in SOP PEPL-6.01.

7. Measure the conditioning environment's temperature and RH on a continuous basis during presampling filter conditioning using electronic instruments whose outputs are recorded by a data acquisition system connected to a computer.

8. Calculate the means and ranges of the temperature and RH over the preceding 24-hour period.

9. Verify that the mean temperature in the conditioning environment has remained within the acceptance criteria (see Table 12-1) and that the temperature during the weighing session is controlled to within ±2 °C.

10. Verify that the mean RH has remained within the acceptance criteria (see Table 12-1) and that the RH during the weighing session is controlled to within ±5%. Record these values in the LIMS and/or in the laboratory QC notebook. If the conditioning environment has not remained within these specifications, the filters cannot be weighed. Take appropriate troubleshooting and corrective actions in that event.

11. If necessary, clean the microbalance’s weighing chamber and the surrounding area with a fine, antistatic brush.

12. Clean the surfaces near the microbalance with disposable laboratory wipes

13. Clean the forceps for handling mass reference standards and the filter forceps with an ethanol-dampened, lint-free cloth or with the premoistened wipes. Allow the forceps to air-dry after cleaning. Make sure that the forceps are thoroughly dry before use because even a small amount of moisture can cause a significant measurement bias.

14. Leave the microbalance plugged in and the power turned on at all times. This procedure enables the microbalance to be operational at any time and eliminates the need for a preliminary warmup period before analyses can be performed. The LCD screen does not need to be on

15. Have all equipment clean and ready to prepare filter cassettes: caps, cassettes, stainless steel screens, 3 x 5" antistatic self-sealing bags, Form BAT-01, and indelible in markers or labels. .

8.1.2 Microbalance-Turning off Default Auto Zero

The Sartorius@ Model MC5's autozero function should be turned off. The factory preset default for this choice is always to have the autozero turned on. Ideally, the Sartorius service technician makes this adjustment when setting up the balance. If this has not been done, follow the procedure on pages 2-2 and 2-6 of the Sartorius Installation and Operating Instructions. The appropriate steps follow to change the autozero menu code setting:

Access the Menu:

7. Turn the balance off; turn it back on again

8. While all the segments are displayed (note a lot of 8s), briefly press the Tare/ key.

9. If -L- is displayed, unlock the menu as follows:
   -Remove the protective cap located on the left-hand side of the balance's electronic computing device's rear panel to expose the menu access switch.
   -Move the switch in the direction of the arrow (toward the right).

Set the code:

7. Press the F1/ key to change the left-hand number to 1.

8. Press the key with the underlined zero with the dot in the center to move to the middle number

9. Press the F1/ key to change the middle number to 6.

10. Press the key with the underlined zero with the dot to move to the right-hand number (When you move to the right-hand number, the previously set numeric code will be indicated- that is, the auotozero code, 161).

11. Press the F1/ key once to change the right-hand number of the code to 2.

Confirm the code setting:

You must press the TARE/ key in order to confirm the code you have just set. This is indicated by the “o" after the code.

1. Press the CF/ key to **store the new menu setting.**

8.2 Presampling Filter Weighing Procedure

This procedure describes the method for presample weighing of a box of filters (~50 filters) that have been properly conditioned. Steps 1- 8 helps warm up the balance with a load, called exercising the balance.

1. Open and close the microbalance draft shield (circular arrow key) two times to equilibrate the air in the draft shield chamber with room air.

2. Zero (using the TARE key) and internally calibrate (using the F1 key) the microbalance according to the microbalance's operating manual. This may take 1-2 minutes.

3. Open the microbalance's draft shield.

4. Using working standard forceps, gently place the 100-mg working mass reference standard on the sample pan. Close the microbalance's draft shield.

5. Wait until the microbalance’s display of the selected unit (mg) of weight indicates that a stable reading has been obtained.

6. After a stable reading is indicated, time 20 seconds and if the weight remains stable, record the measured value of the standard in the LIMS and/or on the Filter Weighing Data Entry Form BAT-01 and go to step 8

7. If the weight fluctuates within this 20 seconds, time another 20 seconds and record the reading. If after three attempts the microbalance does not provide a steady reading, take the weight off and repeat steps 2-5.

8. Repeat steps 3-7 with the 200-mg working standard weight.

9. If the verified and measured values of either working standard disagree by more than ±3 µg, repeat steps 1-8. If the weights are acceptable, move to step 11.

10. If the two values still disagree, halt routine weighing and troubleshoot the entire measurement system and take appropriate corrective action. Corrective action may include:

    - conducting the calibration sensitivity test (Sartorius Installation and Operating Instructions, page 1-39),
    - calibrating the microbalance using the microbalances internal standard
    - recertifying the working standards against the laboratory primary standards, and/or asking the WAM to have a service technician adjust or repair the microbalance (PEPL-7.01)
    - checking temperature and relative humidity with independent standards
    - calibrating the microbalance using an external laboratory primary standard (PEPL-7.02)

11. Shut the draft shield and allow the microbalance to come to zero. Wait at least 20 seconds to ensure zero is achieved. If not achieved by then, the instrument can be manually zeroed by using the TARE key.

12. Select the next routine filter for weighing and record the filter ID located on the outer polyolefin support ring onto Form BAT-01. Indicate the filter type as either “00" for a field filter or “LB” for a laboratory blank

13. Take the filter from its Petrislide by gently pushing down (with the filter-handling forceps on the outer polyolefin support ring) one side of the filter. This should raise the other end of the filter and with a slight nudge, rest the filters edge on the Petrislide. You can then access the outer polyolefin support ring with the filter-handling forceps.

14. Lay each filter, with its support ring side kept upward, on the ^{210}Po antistatic strip for 30 to 60 seconds immediately prior to weighing. Depending on the number of ^{210}Po antistatic strips available, procedures 12-14 can occur with a number of filters.

15. Open the microbalance draft shield.

16. Immediately transfer the filter to the center of the microbalance’s weighing pan and then close the draft shield. Center the filter on the weighing pan.

17. After the microbalance’s display of the selected unit of weight indicates a stable reading, time 20 seconds (as in steps 6 and 7 above) and record this value in the “Weight 1" column on Form BAT-01. If this process takes much longer than 20-30 seconds, evaluate conditions of the weighing room to identify a possible reason

18. Remove the filter and put it back into the Petrislide and set it aside.

19. Take another filter and repeat steps 12-18.

20. After weighing 15 routine filters, or the end of the session, reweigh the first (routine) filter on every Form BAT-01 as a duplicate filter.

21. If the duplicate filter measurement is within ±15 µg of the original weight, go to step 24.

22. If the duplicate filter measurement disagrees from the original measurement by more than ±15 µg, flag the filter "FLD" in the duplicate area. Set it back into the Petrislide and close it. Mark the Petrislide "FLD" and set it aside from the other routine samples.

23. Reweigh the second and third filters on Form BAT-01. If either of these measurements also disagree by >±15 µg, place all samples back into the conditioning environment for a minimum of 12 hours and repeat the weighing procedure. Troubleshoot the entire measurement system and take appropriate corrective action.

24. At the end of the 15 routine weighings, reweigh both working standards by repeating steps 3-5. Compare the verified and measured values of the working standards; they should not disagree by more than ± 3 ug. If unacceptable, repeat steps 1-8. Record the measurements in the LIMS and/or on Form BAT-01. File the Form BAT-01 under SAMP/223.

25. Before weighing another 15 routine filters, follow the procedures in Section 8.2.2.

26. After completing the procedure in Section 8.2.2, repeat steps 3-25 if more filter are to be prepared.

NOTE: Petrislides with these filters should **not** be placed on the same tray that holds filters being conditioned from the field.

8.2.1 Laboratory Blanks

Weigh enough laboratory blanks (5 per filter box of 50 filters) during a presampling weighing session to provide at least one single-use laboratory blank during each subsequent postsampling weighing session. Label the laboratory blank "LB" for filter type.

1. Follow steps 12-19 above.

2. Place the laboratory blank back in the Petrislide.

3. Label the Petrislide with the filter ID and the filter type.

4. Place the laboratory blank back into the filter conditioning environment. Leave it 3/4 covered similar to a normal sample.

8.2.2 Filter Field Preparation

This procedure describes the process for placing the filters that have just been weighed into their cassettes, metal filter caps, and plastic shipping bags for shipment to EPA Regions.

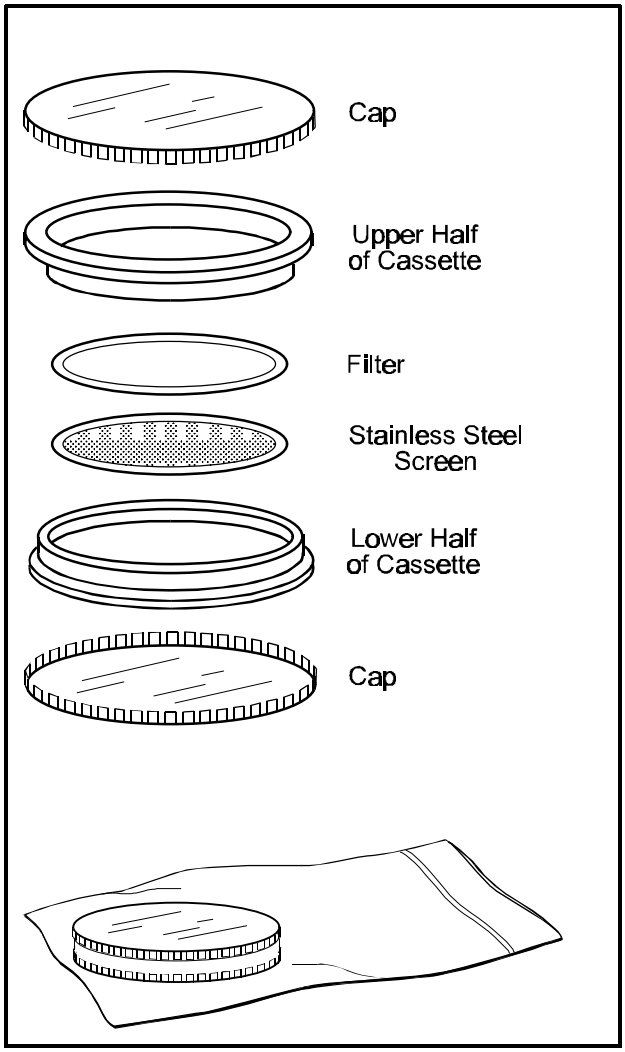

Figure 8.1 Filter cassette equipment and filter cassette in antistatic sample bag.

NOTE: The filter IDs on the filter support ring are very small, which makes them prone to entry errors. These filters should be placed in the same order that they are listed on BAT-01 in order to avoid data entries. However, before placing the filter into a cassette, reverify the filter numbers.

Figure 8.1 illustrates the filter cassette assembly necessary for shipment

1. Select a weighed filter and take a clean filter cassette.

2. Identify the correct filter ID on Form BAT-01.

3. Record the cassette ID onto Form BAT-01; place the filter into the cassette.

4. Record the same cassette ID on a plastic antistatic self sealing shipping bag (label or indelible marker).

5. Place the protective metal filter caps on the cassette, and place the cassette into the antistatic self sealing plastic shipping bag and seal the bag.

6. Select another routine filter and repeat steps 1-5.

7. Add this cassette value and presampling weighing date to a Filter Inventory and Tracking Form COC-01.

8.2 Postsampling Filter Weighing

This procedure describes the method for postweighing filters that have been batched according to SOP PEPL-6.01(section 8.3.1).

1. Follow the postsampling filter handling procedures in SOP PEPL-5.01 and the postsampling filter conditioning procedures in SOP PEPL-6.01(section 8.3.2).

2. Select the appropriate Filter Data Entry Form BAT-01

3. Place the filters to be weighed in the order they are listed on Form BAT-01.

4. Open and close the microbalance draft shield (circular arrow key) two times to equilibrate the air in the draft shield chamber with the room air.

5. Zero (using the TARE key) and internally calibrate (using the F1 key) the microbalance according to the microbalance's operating manual. This may take 1-2 minutes.

6. Open the microbalance's draft shield.

7. Using working standard forceps, gently place the 100-mg working mass reference standard on the sample pan. Close the microbalance's draft shield.

8. Wait until the microbalance’s display of the selected unit (mg) of weight indicates that a stable reading has been obtained.

9. After a stable reading is indicated, time 20 seconds and if the weight remains stable, record the measured value of the standard in the LIMS and/or on Filter Weighing Data Entry Form BAT-01. and go to step 8

10. If the weight fluctuates within this 20 seconds, time another 20 seconds and record the reading. If after three attempts the microbalance does not provide a steady reading, take the weight off and repeat steps 2-5.

11. Repeat steps 6-10 with the 200-mg working standard weight.

12. If the verified and measured values of either working standard disagree by more than ±3 µg, repeat steps 3-11. If the weights are acceptable, move to step 14.

13. If the two values still disagree, halt routine weighing and troubleshoot the entire measurement system and take appropriate corrective action. Corrective action may include:
- conducting the calibration sensitivity test (Sartorius Installation and Operating

Instructions, page 1-39),
- calibrating the microbalance using the microbalances internal standard
- recertifying the working standards against the laboratory primary standards, and/or asking the WAM to have a service technician adjust or repair the microbalance (PEPL-7.01)
- checking temperature and relative humidity with independent standards
- calibrating the microbalance using an external laboratory primary standard (PEPL-7.02)

14. Shut the draft shield and allow the microbalance to come to zero. Wait at least 20 seconds to ensure zero is achieved. If not achieved by then, the instrument can be manually zeroed by using the TARE key.

15. Select a filter for weighing and record the filter ID located on the outer polyolefin support ring onto Form BAT-01. Indicate the filter type as either “RO" for a routine filter, “LB” for a laboratory blank, “FB” for a field blank, “CO” for a collocated sample, “BD” for a batch duplicate, or “PD” for a duplicate from a previous batch.

16. Take the filter from its Petrislide by gently pushing down (with the filter-handling forceps on the outer polyolefin support ring) one side of the filter. This should raise the other end of the filter and with a slight nudge, rest the filters edge on the Petrislide. You can then access the outer polyolefin support ring with the filter-handling forceps.

17. Lay each filter, with its support ring side kept upward, on the ^{210}Po antistatic strip for 30 to 60 seconds immediately prior to weighing. Depending on the number of ^{210}Po antistatic strips available, procedures 16-17 can occur with a number of filters.

18. Open the microbalance draft shield.

19. Immediately transfer the filter to the center of the microbalance’s weighing pan and then close the draft shield.

20. After the microbalance’s display of the selected unit of weight indicates a stable reading, time 20 seconds (as in steps 6 and 7 above) and record this value in the “Weight 1" column on Form BAT-01. If this process takes much longer than 20-30 seconds, evaluate conditions of the weighing room to identify a possible reason

21. Remove the filter and put it back into the Petrislide and set it aside.

22. Take another filter and repeat steps 14-21.

23. After weighing 15 routine filters, reweigh the first (routine) filter on every Form BAT-01 as a duplicate filter.

24. If the duplicate filter measurement is within ±15 µg of the original weight, go to step 27.

25. If the duplicate filter measurement disagrees from the original measurement by more than ±15 µg, flag the filter "FLD" in the duplicate area. Set it back into the Petrislide and close it. Mark the Petrislide "FLD" and set it aside from the other routine samples.

26. Reweigh the second and third filters on Form BAT-01. If either of these measurements also disagree by >±15 µg, place all samples back into the conditioning environment for a minimum of 12 hours and repeat the weighing procedure. Troubleshoot the entire measurement system and take appropriate corrective action.

27. At the end of the 15 routine weighings, reweigh both working standards by repeating steps 6-11. Compare the verified and measured values of the working standards; they should not disagree by more than ± 3 ug. If unacceptable, repeat steps 1-8. Record the measurements in the LIMS and/or on Form BAT-01. File the Form BAT-01 under SAMP/223

28. Review data. In particular, look at the presampling batch form for the field and lab blanks and determine if they meet acceptance criteria (±30 ug and ± 15 ug respectively) if they do not, mark the flag field on the entry form FFB or FLB respectively.

    NOTE: Some filters may lose weight due to volatilization of particulates. It is important to keep the initial weights of these samples.

    NOTE: Keep the routine sample that was used for the duplicate weighing and place it with the next batch. Mark the Petrislide lid with a PD and the postsampling batch number. Do not make this sample one of the first three filters in the next batch.

9.0 Troubleshooting

Problems in meeting the various QC requirements during a presampling or postsampling weighing session can be related to the filter conditioning environment, a malfunctioning microbalance, or the filters themselves (exposed filters).

9.1 Conditioning Environment

- ensure temperature and humidity requirements are within the acceptance criteria. Take corrective action if not
- check temperature and relative humidity monitoring devices with independent devices
- minimize laboratory contamination. More frequent cleaning may be required
- check for static problems and check the polonium strips

9.2 Microbalance

- run the calibration test sequence from p. 1-39, Sartorius Installation and Operating Instructions
- calibrating the microbalance using the microbalances internal standard (F1 Key)
- recertify the working standards against the laboratory primary standards (PEPL-7.01)
- calibrate the microbalance using an external laboratory primary standard (PEPL-7.02)
- have the WAM authorize a service technician to adjust or repair the microbalance. Do not attempt to adjust or repair the microbalance.

9.3 Filters

- recondition and reweigh exposed filters to help determine if they may be composed of nitrates or other particulates that may volatilize and continue to lose weight
- investigate any unused filter whose weight is outside the normal range (i.e., 110 to 160 mg)
- a consistent negative replication (>15 µg) for laboratory blank filters, is usually a sign that the filters have not equilibrated long enough or possibly off gassing of semi-volatiles

PEP Filter Weighing Data Entry Form

Batch Type: PRE POST Batch No. _____

Date _____ Analyst Initials _____

Mean Temp for Past 24 hours: _____SD:_____
Mean RH for Past 24 hours: _____SD:_____

Sample	Filter ID	Filter Type RO/ LB/FB CO/BD/PD	Cassette ID	Weight 1 xxx.xxx mg	Weight 2 xxx.xxx mg	Flag
QC1	100 mg					
QC2	200 mg					
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Routine Filter						
Duplicate 1		BD				
Duplicate 2		DU				
Duplicate 3		DU				
QC1	100 mg					
QC2	200 mg					
BAT-01						

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 9.0 Shipping

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Shipping

SOP: PEPL-9.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP describes the shipping of filters and equipment to field regions.

2.0 Summary of Method

Every two weeks, after the presampling weighing has been completed, filter cassette assemblies, and COC forms are shipped by the LA to the field office. Send enough filters for routine samples, field blanks, and collocated sampling. On a monthly basis, insulated shipping containers, ice substitutes, and maximum-registering thermometers are shipped by the LA back to the field office.

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

4.0 Cautions

Ensure filter shipments fulfill both filter shelf life limitations, site sampling scheduling requirements and shipment timing limitations. See tables 4-3 through 4-4 in PEPL-4.01 for critical filter requirements.

5.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training is required.

6.0 Apparatus and Materials

The following equipment is required to perform this procedure:

- filter cassette assemblies in 3 x 5" antistatic bags
- Filter Inventory and Tracking Form COC-1
- Laboratory Shipment Filter Chain of Custody Record Form COC-2
- PEP Equipment Shipment Tracking Form EQP-01
- Utek -1°C ice substitutes
- maximum/minimum thermometers
- insulated shipping containers
- large (9 x 12") zip-lock bags
- pre-addressed Federal Express Shipping labels
- Federal Express cardboard envelopes
- bubble-wrap packing material
- cardboard shipping boxes (for filter shipping containers).

7.0 Shipping Procedures

7.1 Filter Cassette Assembly Shipping Procedure

This procedure describes the method for shipping filters that have gone through presampling weighing and been assembled in cassettes as described in SOP PEPL-8.01 and the presampling COC procedure in SOP/section PEPL-10.01. Filters will be shipped to each EPA Region every 2 weeks based upon the estimated biweekly requirements for sample and blank filters in Table 9-1. The frequency requirement criteria for the PEP is 1/ sampler/week.

Table 9-1. Filter Preparation/Shipment Requirements

Region 4 Laboratory			**Region 10 Laboratory**		
Region	Monthly Filter Requirement	Biweekly requirement	Region	Monthly Filter Requirement	Biweekly requirement
1	9	5	5	21	11
2	8	4	7	9	5
3	13	7	8	7	4
4	24	12	9	14	7
6	15	8	10	7	4
Total	**69**	**36**	**Total**	**58**	**31**

1. When the filters should be shipped to field personnel, use the Filter Inventory and Tracking Form COC-1 to select the filters.

2. Select the next pre-addressed Federal Express shipping label.

3. Select Filter Chain of Custody Form COC-2 and completely fill in the area “PART I Weighing Lab” information for the shipment. Place Form COC-2 into a 9 x 12" plastic zip-lock bag.

4. Place the selected filter cassette assemblies already in the 3 x 5" bag into the 9 x 12" plastic zip-lock bag with Form(s) COC-2. Include enough blank filters in cassettes to provide 1 field blank per week per portable PEP sampler.

5. Close the 9 x12" plastic bag, fold the bag in half, and wrap it in bubble wrap securing it with a rubber band or tape.

6. Place the 9 x12" plastic bag into an insulated shipping container with enough bubble wrap to prevent excessive movement. Close the insulated shipping container and secure it with shipping tape and ship to the field office.

7. Inform the Regional FS of the shipment per SOP PEPL -4.01.

7.2 Field Equipment Shipping

Sample shipping containers, maximum/minimum thermometers, and ice substitutes need to be returned to the EPA FS once a month or as necessary. In order to ensure that equal supplies are shipped out, the thermometers and shipping containers will be labeled by EPA Region (e.g., R1, R2...).

1. Select a Equipment Shipment Tracking Form EQP-01.

2. Wrap maximum/minimum thermometers separately in bubble wrap and place into a separate shipping container if possible. Add additional bubble wrap to minimize movement.

3. Place the unfrozen -1°C ice substitutes in one shipping container with. For each filter shipping container sent back, include at least 4 ice-substitutes. Add additional bubble wrap to minimize movement.

4. Also send remaining bubble wrap back to Field Office in this shipment.

5. Complete Form EQP-01.

6. Pack the filter shipping containers into a shipping box and include a copy of Form EQP-01 .

7. Ship by UPS or Fed EX

8. Inform the Regional FS of the shipment based on according to SOP PEPL-4.01.

PEP Equipment Shipment Tracking Form							
Analyst ID	Date	Region	# Boxes	# Filter Containers	# Ice substitutes	# Thermometers	Shipment Tracking #
EQP-01							

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 10.0 Filter Chain of Custody

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Filter Chain of Custody

SOP: PEPL-10.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This procedure describes all the procedures necessary for ensuring that:

- filters are processed, transferred, stored, and analyzed by authorized personnel
- sample integrity is maintained during all laboratory phases of sample handling and analyses
- an accurate written record is maintained of sample handling and treatment from the time of receipt from EPA through laboratory procedures to disposal

Proper sample custody minimizes accidents by assigning responsibility for all stages of sample handling and ensures that problems will be detected and documented if they occur. A sample is in custody if it is in actual physical possession or if it is in a secured area that is restricted to authorized personnel.

2.0 Summary of Method

Filter chain of custody (COC) starts as soon as filters are preweighed, when filter weights and unique filter ID numbers are initially recorded. Although initial COC information is recorded by hand, the information will be entered into the sampling tracking system, where an electronic record will be kept. This section addresses sample custody procedures at the following stages:

- Presampling,
- Postsampling, and
- Filter archive.

This SOP does not cover field chain of custody COC procedures, which are discussed in the field SOPs. The PEP will use flags and free-form notes to help describe the quality of the data. COC forms have a flag area where multiple flags can be entered. The flags and their definitions can be found in Appendix B.

3.0 Definitions

3.1 Acronyms

Acronyms and abbreviations are listed in the front of this document.

3.2 Forms

The following forms are found at the end of this SOP:

- Filter Inventory and Tracking Form COC-1
- PEP Chain of Custody Form COC-2
- Filter Archiving Tracking Form COC-3

4.0 Cautions

The most important variables in the COC activity are the filter number and the cassette ID. The unique filter ID number on the filter support ring is small, and care must be taken to read this number correctly. Since the filters will be placed into the filter cassettes at preweighing, the cassette ID will be used extensively during COC procedures. Care must be taken in reading and recording this number.

Most fields on the COC forms should be completed. The LA should ensure that all mandatory fields are completed.

Filters must always be handled carefully. During postsampling inspection, do not turn the filter upside down, or jostle it.

5.0 Apparatus and Materials

The following equipment is required to perform this procedure :

- laboratory coat
- powder-free laboratory antistatic gloves
- COC notebook
- filter trays
- COC forms
- filters (preweighed and ready for shipping)
- flag definitions
- Petrislides
- filter ID labels
- Filter Weighing Data Entry Form BAT-01

6.0 Sample Custody Procedures

The LA is designated as the sample custodian and will be responsible for sample custody during all laboratory data operation stages. Initials of the sample custodian will be used on all COC forms. An alternate sample custodian may be designated but he or she must be trained, certified, and approved by the WAM.

6.1 Presampling Custody

This section contains a description of the COC procedures for filters after they have undergone presampling weighing procedures. Section 8.1, SOP PEPL-8.01, defines how the filters will be enumerated, weighed, and placed into cassettes, filter caps, plastic antistatic shipping bags (labeled with the cassette number), and stored prior to shipment to field sites. The LA must remember the following:

- filters must be used within 30 days of preweighing
- filter cassette assemblies in the 3 x 5" cassette bags that have been preweighed will be moved to the designated field filter shelf in order to separate them from filters being conditioned
- Filter Inventory and Tracking Form COC-1 containing the filter ID, the cassette ID, and the preweighing sampling date will be attached to the field filter shelf for use by the LA

Every 2 weeks the LA will select the appropriate number of filters to send to the field scientists (see Table 9-1 in SOP PEPL-9.01).

1. Put on a laboratory coat.

2. Select a Chain of Custody Form COC-2 for each filter being shipped to a Region.

3. Review Filter Inventory and Tracking Form COC-1 and select the next set of filters on the sheet that will be needed for each Region, based on Table 9-1. Date and initial Form COC-1.

4. For each Form COC-2, completely fill out the section “Part 1 Weighing Laboratory” and initial. Make sure you fill in the area “This Filter Must be Used By” by adding 30 days to the preweighing sampling date of each cassette.

5. Tear off the last copy of COC-2 for the lab analyst records

6. Pack filters for travel to the field using SOP PEPL-9.01. And include the COC-2 forms for each filter.

NOTE: A copy of the Form COC-2 is required to be shipped back to the laboratory with the filters. The lab analyst will review the forms using the procedures in Section 6.2. below.

6.2 Postsampling Custody

The field sampling SOPs specify the techniques for properly collecting and handling the sample filters and the COC requirements to ship filters and processing back to the laboratories. Following are the COC procedures required for receiving these sample filters.

6.2.1 Filter Receipt Procedure

The samples, whether transported to the laboratory by the field scientists or by next-day air, will be received by the LA or a certified alternate.

1. Receive the shipping/transport container(s).

2. Upon receipt, open the container(s) to find the filter cassettes, the chain of custody form (s) COC-2 , the Field Data Sheet and the portable sampler data diskette(s).

3. Check the forms to ensure they are completely and appropriately filled out. Ensure dates are appropriate (i.e., removal date same or earlier than date shipped).

4. Store the diskette in the diskette folder by Region. See SOP PEPL-11.01

5. Fill out “Part V Weighing Laboratory” of the Form COC-2. This will be the initial check of the cooler temperature and to ensure that the number of filters shipped were received.

NOTE: Depending upon whether the filters that have been received will be used in the next batch analysis, they may either be placed in a refrigerator to maintain them at 4 ^{O}C or in the weighing room conditioning environment. **Filters should not be inspected until they have been allowed to equilibrate to weighing room temperature for a minimum of 12 to 24 hours.**

NOTE: Label and position presampling and postsampling filters in a manner that distinguishes and separates them.

6. Place the exposed filter cassette assemblies in the 3 x 5" cassette bags on a filter tray with the appropriate Form COC-2 in a plastic 9 x 12" zip-lock bag.

7. Place the exposed filter cassette assemblies in the 3 x 5" cassette bags in the refrigerator and follow step 8 or in the conditioning environment and follow step 9.

8. When ready to weigh a batch (SOP PEPL-8.01) of postsample filters, take the appropriate filters out of the refrigerator.

9. After at least 12 hours of thermal equilibration, start the filter inspection process.

10. Inspect the antistatic plastic cassette bag to ensure that the cassette ID/filter type is recorded on the bag and matches the cassette ID/filter type on part I and III of COC-2. If the values do not match or if the filter type is not included on the plastic bag, place an "EER" flag in the flag area and take corrective actions by rectifying errors with the field scientist. Enter the flag explanation in the Free Form Notes area.

11. Take the filter cassettes out of the 3 x 5" plastic cassette bag (keep the bag) and take the metal filter caps off the filter cassette.

12. Check to ensure that the cassette number on the plastic cassette bag and on the cassette match. Once the match is confirmed, the plastic bag can be discarded. If the values do not match, place an "EER" flag in the flag area and take corrective actions by rectifying errors with the field scientist. Enter the flag explanation in the Free Form Notes area of COC-2

13. Place on powder free gloves and remove the filter from the filter cassette.

14. Examine the Filter Integrity Flag column on Part III of COC-2. If the integrity is marked "NO" examine the filter and add any additional free-form notes. If it appears that the sample is damaged or the integrity is questionable, place a "SIS" flag in the Part V flag area

15. Inspect the filter in a manner similar to initial filter integrity examination without using a light table but looking for obvious damage that would affect the quality of the data. Do not flip or turn the filter over. If the filter appears to be damaged and that damage is not explained in the Postsampling Filter Recovery area or Free-Form Notes area, place a "NO" in the "Received Condition OK" an add free form notes on the problem.. If the filter appears to be in good shape, place a "YES" in the "Received Condition OK" area.

16. Place the examined filter in a Petrislide that is labeled with the filter ID and filter type. As carefully as possible, check to ensure that these values match.

17. Repeat steps 10-16 for each filter.

18. Place completed Form COC-2 into the COC notebook

19. The filters will now remain in the filter weighing room for conditioning and postsampling weighing activities.

6.3 Filter Archive

Upon completion of postweighing activities, Filter Archive Tracking Form COC-3 is used by the LA to archive the filter. Each filter is placed in the labeled Petrislide and stored in a box uniquely identified by program, year (four digit year), and box number (two digits). Samples are archived in the refrigerator for 1 year past the year of collection and at ambient conditions for two additional years. Prior to disposal, the archivist's organization should notify OAQPS of the intent to dispose of the filters.

7.0 Corrective Actions

It is important that all filters that have been preweighed and shipped to the field can be tracked through all stages of data collection to ensure that they are processed within appropriate time frames and that their sample custody and integrity are confirmed. During shipping and receiving procedures, data may be incorrectly entered or integrity flags may indicate problems in sampling, handling, or shipping. The following procedures are used to take corrective action.

7.1 Suspected Entry Errors

1. Contact the field scientist by e-mail and send copies to the laboratory and the field WAM.

2. Explain what appears to be the entry errors and ask for recertification. Save a hardcopy of the e-mail for records.

3. Upon receipt of the response, make appropriate corrections. Save the response and add it to the e-mail.

4. Include corrective action information in the weekly progress report.

7.2 Integrity flags-

These flags are associated with damaged filters or filters of questionable integrity.

1. A listing of the filters that are flagged will be presented to the WAM.

2. The WAM will decide to stop any additional processing on the filters. If this is the case, a "VOD" (void) flag will be included in Part V the Shipping Integrity Flags column Form COC-2.

3. The WAM may decide to allow processing of filters and determine the validity of data after reviewing the concentration information or the comparisons to the routine data.

8.0 Records Management

All completed COC forms will be placed into the COC notebook. This notebook will be filed under TRAN/643 in the reporting package file.

PEP Filter Archiving Tracking Form

Filter ID	Analysis Date	Archive Date	Box ID/Box #	Archived By	Comments

COC-3

$PM_{2.5}$ Federal Reference Method Performance Evaluation Program
Chain of Custody Form

PART I - WEIGHING LABORATORY

Filter Weighing and Shipping Information			
Filter ID Number		Filter Cassette No	
Weighing Lab		Cassette Type	
Analyst/Custodian Name		Weighing Date	
Shipment Date		Airbill No.	
Sent to (PE Org)		Shipped via	Fed. Express
This Filter Must be Used by:		Return to:	

On completion of Part I, the weighing laboratory keeps one copy and sends 2 copies to the field office with the filter.

PART II - FIELD OFFICE

Date Received:		PE Organization:	
Shipment Integrity OK?	**Q** Yes **Q** No (describe)	Field Scientist:	

PART III FIELD SITE

Filter Type				
Q RO	**Q** CO	**Q** FB	**Q** Void (describe)	**Q** Other (describe)
Associated Filter Samples - *enter cassette numbers for other filters used for this exposure*				
PE Sample	Colloc. PE	Field Blank	Other (describe)	Other (describe)

Transport of Filter and Field Site Information				
Arrival Date at Site:		Site Name:		
AIRS Site ID:		Primary Site Sampler:	Make/Model:	Ser. No:
Site Operator and Other Observers:				
Filter Integrity OK	**Q** Yes **Q** No (describe)			

PART IV FIELD FILTER SHIPPING

Shipping from Field to Weighing Lab					
Shipped by:		Shipment Date:		Shipped via:	Fed.Express
Airbill No.		Destination:			

On completion of Part II-IV, the field scientist keeps one copy and sends the other to the laboratory with the filter.

PART V - WEIGHING LABORATORY

Received by:		Date Received:		Integrity Flag:
Received Condition OK?	**Q** Yes **Q** No (describe)	Max Temperature:	°C	Cold Pack Condition: **Q** frozen **Q** cold **Q** ambient

Notes:

Filter Inventory and Tracking Form

Filter ID	Cassette ID	Presample Weigh Date	Pre-sample Shipping Date	Region	Sample Start Date	Sample End Date	Laboratory Receipt Date	Postsample Weigh Date	AIR Upload Date	Flags	LA Initials
FORM COC-1											

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 12.0 Quality Assurance/Quality Control

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Quality Assurance Quality/Control Summary

SOP: PEPL-12.01

Name: Printed	Signature	Date
Mark Shanis		

Contents

1.0 Scope and Applicability

This SOP describes quality control (QC) activities in the weighing laboratory of the $PM_{2.5}$ FRM PEP, which provides quality assurance (QA) activity support to the $PM_{2.5}$ monitoring community.

2.0 Summary of Method

Record all QC data in the LIMS and/or the laboratory QC notebook, including the microbalance calibration information, working standard verifications, routine internal QC checks of working standards and laboratory and field filter blanks, duplicate filter measurements, and internal microbalance PEs. The forms for the initial collection and summarization of these QC data are found in the preceding SOPs.

These data may duplicate data that are already recorded in the LIMS and/or in equivalent paper laboratory data forms, but they will be consolidated so that long-term QC data trends can be identified. Maintain control charts for each microbalance and include these charts in the LIMS and/or in an equivalent laboratory QC notebook. These charts may allow the discovery of excessive drift or imprecision that could signal an instrument malfunction.

Table 12-1 summarizes the acceptance criteria and calibration frequencies for weighing laboratory QC checks.

Table 12-1 Acceptance Criteria for Laboratory QC Checks

Requirement	Measurement Frequency	Acceptance Criterion	QA Handbook Document 2.12
Conditioning Environment			
Temperature	Each weighing session	24-hour mean temp between 20 and 23° C, control ± 2° C (SD)	2.12 Sec. 7.6
Relative humidity	Each weighing session	24-hour mean RH between 30% and 40% RH, control ± 5% RH (SD)	2.12 Sec. 7.6
Filter Blanks			
Lot blanks	3/box,3 boxes/lot,9 total (Daily for at least 5 days)	max diff of ±15 μg/5days, 5μg/day/batch(of 2-3)	2.12 Sec. 7.6
Lot Exposure blanks	1pre-,1postsampling weighing session/batch	avg diff of 3, not>5μg , no filter diff >15μg	Not described
Lab blanks	One per weighing session	±15 μg difference	2.12 Sec. 7.8
Field blanks	One per sampler/wk	±30 μg difference	Not described
Calibration and Verification			
Working standards verification	Quarterly	±2 μg	2.12 Sec. 7.3
Mass standards calibration	Yearly	Not applicable	2.12 Sec. 7.3
Balance calibration	Semiannually or as needed	Not applicable	Not described
Temp/humidity verification	Quarterly	± 2°C/±2%	Not described

Requirement	Measurement Frequency	Acceptance Criterion	QA Handbook Document 2.12
Microbalance QC Checks			
Working standard QC check	Start and end of each weighing session	±3 μg	2.12 Sec. 7.8
Duplicate filter weighing	1 filter at end of weighing session; 1 carried over to next session with lab blank	±15 μg	Not described
Performance Evaluations			
Interlaboratory Comparisons	1/quarter	TBD	Not described

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

4.0 Personnel Qualifications

Certification by passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ training is required.

5. Materials Used in PEP Laboratory QC

5.1 Filters - Lot Blanks, Lot Exposure Blanks, Laboratory Blanks, and Field Blanks

Four types of blank filters are used in the PE program.

Lot blanks, representing a new lot or shipment of filters, are unconditioned, unsampled filters used to determine if effects on filter weight stability have occurred due to the volatilization of material from the filter or to the absorption of gaseous material into the filter from the atmosphere.

Lot Exposure Blanks are blank filters from a new lot that are part of a batch of filters that will be conditioned together before weighing, and are included to show the effects of any event associated with a particular conditioning period of laboratory exposure. These blanks are only used with the batch of filters with which they are conditioned.

Laboratory blanks are conditioned, unsampled filters used to determine any weight change between presampling and postsampling weighings due to contamination in the microbalance and conditioning environment.

Field blanks are conditioned, unsampled filters used to determine whether similar contamination occurs during sampling. Lot exposure and field transport blanks may also be used if the results of the other QC materials indicate that they are needed to determine sources and causes of

variability. In addition to these blanks, 2-3 loaded filters that have already been weighed after sampling exposure are weighed again in each weighing session to indicate the mass-related effects of the laboratory environment on the sample load as distinguished from the effects on the filter.

5.2 Weights - Working and Laboratory Primary Reference Standard

Use ASTM Class I 5g, 100mg and 200mg weights as the laboratory primary reference standard weights. Also use a set of working standard weights that includes the 5g, 100mg, 200mg, and 5mg reference standard weights. The 100 and/or 200mg weights are used at the beginning and end of each weighing session. The ASTM Class weights are used to verify quarterly that the value of the weights determined by weighing on the microbalance is correct.

6.0 Procedures for Collecting the Laboratory QC Data

6.1 Filters

Measure the weight stability and time to achieve weight stability of each new shipment of filters by opening three new boxes from each shipment and randomly selecting 3 filters from each box to be used as lot blanks. A filter lot is defined as a single shipment of filters from EPA, a filter manufacturer, or other source of filters. After an initial 24-hour conditioning, reweigh these filters periodically (daily for at least 5 days) and store them in the conditioning environment between weighings. Record these measurements in the LIMS and/or in the laboratory batch data form. Continue these weighings until the average weight change of the 9 filters is less than 5μg/day and ±15 µg. This filter weight stability measurement will determine the lot conditioning period over which each filter in the lot must be conditioned before it can be used for routine sampling. Repeat this measurement whenever a new lot of filters is received.

Keep laboratory blanks inside the conditioning environment. Weigh enough laboratory blanks during a presampling weighing session to provide at least one single-use laboratory blank during each subsequent postsampling weighing session (1 per batch; estimating weekly sessions, at least 4 per month; more if more batches are analyzed). (Compare filter batching in Section 8.3.1 in SOP PEP 6.01.) Record the presampling and postsampling weights in the LIMS and/or in the laboratory batch data form. If the weight change in the laboratory blank exceeds ±15 µg, contamination in the conditioning environment may be occurring. Take appropriate troubleshooting and corrective actions in that event. Lab blanks are weighed with pre- and postsampling shipment batch of routine filter weighings.

Send one field blank with each PE sampler/week to the evaluation site. At the evaluation site, it will be momentarily installed in the PE sampler, removed, and stored in its protective containers inside the PE sampler's case at the evaluation site until the PE sample is retrieved for shipment back to the weighing laboratory. Weigh enough field blanks during a presampling weighing session to provide one single-use field blank for each PE sampler/week. Record the presampling

and postsampling weights in the LIMS and/or in the laboratory batch data form. If the weight change in the field blank exceeds ±30 µg, contamination during transportation or at the evaluation site may be occurring. Take appropriate troubleshooting and corrective actions in that event.

Weigh of at least 1 routine filter sample a second time at the end of each weighing session, just before the reweighing of the working standards. Place this sample into the next weighing session batch. If a weight difference > 15μg occurs, troubleshoot the contamination potential of the room as indicated by the blanks; if this does not seem to be the problem, trouble shoot for microbalance zero or calibration drift.

6.2 Balance Calibration Verification QC

Verify the calibration of the microbalance during each weighing session. Weigh two working standards (e.g., 100 and 200 mg) at the beginning and end of the weighing session. Bracket the estimated mass of an unexposed or exposed filter with the masses of these two working standards. If the verified and measured values of a working standard disagree by more than ±3 µg (i.e., three times the microbalance's repeatability), reweigh both working standards. If the disagreement persists, investigate the microbalance's calibration and the working standards and take appropriate corrective action, which may include (1) recalibrating the microbalance using an internal laboratory primary standard, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having an authorized microbalance service technician adjust or repair the microbalance. Verify working standards against the laboratory primary reference standard weights quarterly. Estimate sensitivity during initial microbalance set-up in the weighing room using the 5mg weight.

7.0 Procedures for Collecting Method QC

7.1 Sampling and Weighing Method QC

Weigh the laboratory and field blanks throughout the weighing session. Prepare enough laboratory blanks during a presampling weighing session to provide at least one laboratory blank during each subsequent postsampling weighing session. Weigh enough field blanks during a presampling weighing session to provide at least one single-use field blank for each PE sampler per week. Reweigh both working standards at the beginning and end of each filter weighing session.

Record the working standard, blank, and duplicate filter measurements in the LIMS and/or in an equivalent laboratory data form and the laboratory QC notebook. If the working standard measurements differ from the verified values or the presampling values by more than ±3 µg (i.e., three times the microbalance's repeatability), repeat the working standard measurements. If the laboratory blank or replicate measurements differ from the presampling values or previous postsampling values by more than ±15 µg (i.e., three times the precision for unexposed filters),

repeat the laboratory blank or replicate measurements. If the pre- and postsampling weights for the field blanks disagree by more than ±30 µg, repeat the field blank measurements. If the two working standard measurements--the two laboratory blank measurements or the two field blank measurements--still disagree, troubleshoot the entire measurement system and take appropriate corrective action.

Do not correct PE sample measurements to account for laboratory or field blank measurements. Do not automatically invalidate PE sample measurements because of high blank values that were measured during the same weighing session. If high blank values are discovered, troubleshoot the entire measurement system and take appropriate corrective action to reduce the blank values to acceptable levels.

If more than one microbalance is used, make the pre- and postsampling measurements of each filter on the same microbalance.

The EPA WAM will certify the data in the LIMS and/or in the laboratory data forms as to the acceptability of gravimetric filter analyses and the QC checks and to the completeness of the data. The WAM will sign or initial each completed data form. Bind these forms together to serve as a laboratory data notebook.

8. QA/QC Data Preparation and Evaluation in Control Charts

Use control charts to provide a graphical means of determining whether the various components of the measurement system are in statistical control. Use property charts that graph individual measurements of a parameter (e.g., microbalance QC check or working standard verification) or a mean of several measurements. Table 12-2 indicates which laboratory QC check parameters are control-charted. The control charts are used as an early warning system to evaluate trends in measurement system precision and bias. See (or insert here) the examples of simple zero and span control charts in the new Volume II, Part I, Section 12 of the EPA QA Handbook, page 9 of 13.

Table 12-2. Laboratory QC Check Parameters

Laboratory QC Check	Parameter Plotted by Control Chart
Temperature and RH in the conditioning environment	Mean temperature, temperature range temp standard deviation, mean RH and standard deviation, and RH range for 24 hours immediately preceding each weighing session
Lot blanks	Weekly weight change and lot conditioning periods
Laboratory blank	Difference between initial and final weights by session
Field blanks	Difference between initial and final weights by sampler
Working standard QC check	Difference between measured and verified weights
Duplicate filter weighing	Difference between initial and final weights

9. Performance Evaluations

9.1 Microbalance Performance Evaluations

Conduct an internal PE of each microbalance used to weigh $PM_{2.5}$ filters on an annual basis.

Note: The assessor is independent of the LA . See Figure 1.1 in Section. 1. Use an independent set of ASTM Class 1 mass reference standards for the PE performance evaluation. These weights must be traceable to NIST, with a tolerance of no more than 0.025 mg. Individual weights of 100 and 200 mg are suggested. Do not use the same weights for the PE that are used for the day-to-day calibration verifications of the microbalance, but these weights may be traceable to the same laboratory primary standards that are used to verify the working standards.

Because microbalances are extremely delicate instruments and should not be operated by inexperienced personnel, it is recommended that the PE of the filter-weighing process be done in cooperation with the laboratory personnel. The analyst normally performing the weighings for $PM_{2.5}$ monitoring should assist the assessor by preparing the balance as if a series of filter weighings were to be conducted.

Record all PE data in the LIMS or in the laboratory QC notebook. The balance display should agree with the certified value of the PE weight to within ±20 µg (twice the individual tolerance for ASTM Class 1 standards).

Many laboratories maintain an agreement with a service representative to conduct regular servicing of the balances. The PE program laboratories have arranged to have semiannual calls. It may be instructive to conduct a PE prior to the periodic servicing and again immediately after the servicing.

Should the assessor wish to conduct a technical systems audit (TSA) of the weighing laboratory operations, assess the following items:

1. Review the process by which the laboratory receives samples from the field and how the samples are inspected, checked for temperature, logged in, and conditioned in preparation for weighing. Also review records to ensure that the balance room temperature and the RH have been held within specifications for the last several months.
2. Review the maintenance and calibration log for each balance. Routine balance maintenance and calibrations must be performed by the manufacturer's service representative at manufacturer-specified scheduled intervals. In no case should the interval between service calls exceed 1 year.
3. Review QC data records for the filter-weighing process. Ensure that the following QC activities have been performed and documented:

- working standards checks at the beginning and end of every filter weighing session,
- exposure lot blank checks with the pre and postsampling weighing session for that batch of filters from one filter shipment lot,
- laboratory blank filter weighings every day of balance operation,
- field blank weighings, and
- duplicate filter weighings of 2-3 filters in 1 weighing session, followed by 1 replicate carried-over to the next weighing session).

If any QC checks were out of limits, note what corrective action was taken.

4. Observe the analyst's technique and review the laboratory weighing procedure for determining both the tare and gross weights of the sampling filters. The following scheme can be used to assess technique and weight stability of exposed and unexposed filters:

   - Select randomly and have the analyst reweigh 4 equilibrated unexposed filters out of every group of 50 or less from the most recent group of filters weighed. For groups of 50 to 100, reweigh 7 from each group.
   - Record the original values and the audit weights on an audit form or in a notebook. Calculate the weight difference between the original weight, in mg, and the audit weight, in mg, for each filter.

   For unexposed filters, the difference should be less than ±15 µg (0.015 mg). For exposed filters, the potential loss of volatile particles prevents establishment of meaningful acceptance or rejection limits. Forward the audit data to the laboratory supervisor for review.

5. Review the verification records and the traceability of standards for mass, temperature, and RH.
6. Review control charts for temperature, RH, blanks, working standard QC checks, and duplicate filter weighings.

9.2 Interlaboratory Filter Exchanges

Every quarter, a batch of filter samples that have been postweighed and the data accepted, will be exchanged between the two national laboratories, as soon after the postweighing as possible. These batches will then be sent to the OAQPS laboratory for weighing. This program is still under development as to the required acceptance criteria.

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Section 13.0 Filter Storage and Archiving

Weighing Laboratory Standard Operating Procedure for the $PM_{2.5}$ FRM Performance Evaluation Program

Operation: Filter Storage and Archiving

SOP: PEPL-13.01

Name: Printed	Signature	Date
Mark Shanis		

Contents
(applicable to this SOP)

1.0 Scope and Applicability

This SOP describes the storage of data and of exposed filters after postsampling weighing.

2.0 Summary of Method

Upon completion of postweighing activities, Filter Archive Tracking Form COC-3 is used by the LA to archive the filter. Each filter is placed in the labeled Petrislide, capped, and stored in a box uniquely identified by program, year (four digit year), and box number (two digits). Samples are archived in the refrigerator for 1 year past the year of collection and at ambient conditions for two additional years or until EPA indicates that they can be discarded. Prior to disposal, the archivist's organization should notify OAQPS of the intent to dispose of the filters.

3.0 Definitions

Appendix A contains a glossary of the terms used in the PEP.

4.0 Personnel Qualifications

Certification of passing the written examination and the hands-on practical examination for the laboratory component of $PM_{2.5}$ FRM PE training is required.

5.0 Apparatus and Materials

5.1 Filter Storage and Archiving

- weighed, postsampling filters in labeled Petrislides
- original Petrislide boxes (contains 100 Petrislides/box)
- barrier wrapping or container(such as bubble-wrap or Zip-Lock Bags, big enough to contain the boxes (6" x 6" x 5")
- refrigerator, 4°C or less, frost-free, large enough for 1 year load **(6 boxes)**
- shelf or rack for 2nd and 3rd yr storage of boxes of filters in Petrislides

5.2 Paper Storage and Archiving

- bookcase or file cabinet
- binders, labels; file folders and file hangers

5.3 Electronic Storage and Archiving

- space in existing EPA electronic storage facility or rack/shelf
- diskette organizer boxes and labels

5.4 Facility Requirements

- approx. 4'x12' of room in env.controlled facility; EPA electronic backup in Reg 4 and 10.

6.0 Postsampling Filter Storage and Archive

6.1 Requirements

1. Store the filters after their postsampling weighing in packages of labeled Petrislides for at least 3 years or until EPA indicates that they can be discarded.

2. For the first year, store in refrigerator at 4°C or less; contain labeled packages in vapor barrier such as Ziplock bags.

3. For the second and third years, store the packaged filters in an area that is free from excessive heat, humidity, and contamination. It is not necessary to store them in the conditioning environment.

4. The packages should be labeled with an ID number that is associated with the ID numbers of the filters they contain. Store them in an orderly way that allows for their retrieval when necessary. The LA and WAM should retain cumulative paper and electronic copies.

6.2 Procedure for Filter Storage and Archiving

1. The LA puts the weighed, postsampling filters in their labeled Petrislides in the sleeves in an empty Petrislide box and keeps the sleeves in the box in the conditioning room until the 4 sleeves, 25 slides per sleeve, are all full.

2. The LA labels the box with the next ID number in the box ID number sequence (4- or 10-99-1,4- or 10-99-2, etc.). The LA puts the Box ID number on the filter tracking Archive forms (COC-3) associated with the filters in the Petrislides in the box.

3. When the box is full, the LA delivers the box and associated, partially completed COC-3 forms to the designated archive custodian (may or may not be the same person).

4. On receiving a box, the custodian records, in the appropriate columns on form COC-3, refrigerator location (in comments column), receipt date, and archive custodian name.

5. On transferring box from refrigerator to ambient storage, custodian records transfer date and his/her initials.

6. On final disposition, custodian records date and initials on form COC-3.

7. The custodian keeps the COC Forms (and a cumulative chronological inventory of all the box ID numbers) in a labeled file cabinet and/or computer file. The cabinet is in a location known and at least indirectly accessible to the EPA Region 4 and 10 Lab WAMs.

8. Any removal must be authorized on a form with signature approval of the appropriate Lab WAM and be recorded in on that form, which also contains the signature of the custodian, the date of removal and return, the signature of the remover, and the reason for the removal. The custodian keeps the removal/return forms with the appropriate COC-3 forms.

6.3 Archive Stability Testing

1. The LA provides will identify at least 3 blanks and 3 routine filters. During the first year, the filters should be retrieved by the LA and conditioned and weighed according to the appropriate SOPs in this Compendium (5.01, 6.01, and 8.01) and their weights tracked on control charts. This procedure will develop QC data which can be used to alter procedures establish control limits, and/or characterize the effect of the archive treatment on the filter weights

7.0 Data and Records Management

$PM_{2.5}$ information will be included in the PEP laboratory record and filing system. It is organized in a similar manner to the EPA's records management system and follows the same coding scheme in order to facilitate easy retrieval of information. Table 13-1 includes the documents and records that will be filed. In order to archive the information as a cohesive unit, all $PM_{2.5}$ information should be filed under the major code "PEP," followed by the codes in Table 13-1. Attach a copy of COC-3 to this SOP. Note (Add) the titles End Refrigeration Date and Final Disposition Date, with blanks for completion, above the comments column, in the header space.

Table 13-1. $PM_{2.5}$ PEP Laboratory Filing System

Categories	Record/Document Types	File Codes
Management and organization	Organizational structure	ADMI/106
	Personnel qualifications and training	PERS/123
	Training certification	AIRP/482
	Quality management plan	AIRP/216
	Document control plan	ADMI/307
	EPA directives	DIRE/007
	Grant allocations	BUDG/043-CONT/003
	Support contract	CONT/202
Environmental data operations	QA project plans	PROG/185
	SOPs	SAMP/223
	Laboratory notebooks	SAMP/502
	Communication	SAMP/502
	Sample handling/custody records	TRAN/643
	Inspection/maintenance records	AIRP/486

Categories	Record/Document Types	File Codes
Raw data	Any original data (routine and QC data) including data entry forms and temp and humidity	SAMP/223
Data reporting	Air quality index report Annual SLAMS air quality information Weekly progress reports Data/summary reports Journal articles/papers/presentations	AIRP/484 AIRP/484 AIRP/484 AIRP/484 PUBL/250
Data management	Data algorithms Data management plans/flowcharts $PM_{2.5}$ data Data management systems	INFO/304 INFO/304 INFO/160 - INFO/173 INFO/304 - INFO/170
Quality assurance	Good laboratory practices Network reviews Control charts Data quality assessments QA reports System audits Response/corrective action reports	COMP/322 OVER/255 SAMP/223 SAMP/223 OVER/203 OVER/255 PROG/082 OVER/658

Appendix A

Glossary

The following glossary is taken from two documents: 1) and 2) *EPA Guidance For Quality Assurance Project Plans EPA QA/G-5* as well as additional terms used in the PEP Lab SOPs

Glossary

Acceptance criteria — Specified limits placed on characteristics of an item, process, or service defined in requirements documents. (ASQC Definitions)

Accuracy — A measure of the closeness of an individual measurement or the average of a number of measurements to the true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; the EPA recommends using the terms *"precision"* and *"bias"*, rather than "accuracy," to convey the information usually associated with accuracy. Refer to *Appendix D, Data Quality Indicators* for a more detailed definition.

Activity — An all-inclusive term describing a specific set of operations of related tasks to be performed, either serially or in parallel (e.g., research and development, field sampling, analytical operations, equipment fabrication), that, in total, result in a product or service.

Assessment — The evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation (PE), management systems review (MSR), peer review, inspection, or surveillance.

American National Standards Institute (ANSI)- Administrator and coordinator of the U.S. private sector voluntary standardization system.

American Society for Testing and Materials (ASTM) -A professional organization that develops and distributes protocols for testing and provides reference standards.

Analyst - A staff member who weighs the new and used filters and computes the concentration of $PM_{2.5}$ in $\mu g/m^3$.

ANSI/ASTM Class 1 standards -The standards for weighing operations with a microbalance that are certified by their manufacturer as being in conformance with ASTM's standard specification for laboratory weights and precision mass standards (E 617-9) and particularly the Class 1 specifications. These standards are traceable to NIST.

Audit of Data Quality (ADQ) — A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Audit (quality) — A systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives.

Authenticate — The act of establishing an item as genuine, valid, or authoritative.

Bias — The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). Refer to *Appendix D, Data Quality Indicators,*for a more detailed definition.

Blank — A sample subjected to the usual analytical or measurement process to establish a zero baseline or background value. Sometimes used to adjust or correct routine analytical results. A sample that is

intended to contain none of the analytes of interest. A blank is used to detect contamination during sample handling preparation and/or analysis.

Calibration — A comparison of a measurement standard, instrument, or item with a standard or instrument of higher accuracy to detect and quantify inaccuracies and to report or eliminate those inaccuracies by adjustments.

Calibration drift — The deviation in instrument response from a reference value over a period of time before recalibration.

Cassette - A device supplied with $PM_{2.5}$ samplers to allow a weighed Teflon® filter to be held in place in the sampler and manipulated before and after sampling without touching the filter and to minimize damage to the filter and/or sample, during such activities

Certification — The process of testing and evaluation against specifications designed to document, verify, and recognize the competence of a person, organization, or other entity to perform a function or service, usually for a specified time.

Chain of custody — An unbroken trail of accountability that ensures the physical security of samples, data, and records.

Characteristic — Any property or attribute of a datum, item, process, or service that is distinct, describable, and/or measurable.

Check standard — A standard prepared independently of the calibration standards and analyzed exactly like the samples. Check standard results are used to estimate analytical precision and to indicate the presence of bias due to the calibration of the analytical system.

Collocated samples — Two or more portions collected at the same point in time and space so as to be considered identical. These samples are also known as field replicates and should be identified as such.

Comparability — A measure of the confidence with which one data set or method can be compared to another.

Completeness — A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Refer to *Appendix D, Data Quality Indicators,* for a more detailed definition.

Computer program — A sequence of instructions suitable for processing by a computer. Processing may include the use of an assembler, a compiler, an interpreter, or a translator to prepare the program for execution. A computer program may be stored on magnetic media and referred to as "software," or it may be stored permanently on computer chips, referred to as "firmware." Computer programs covered in a QAPP are those used for design analysis, data acquisition, data reduction, data storage (databases), operation or control, and database or document control registers when used as the controlled source of quality information.

Conditioning environment -A specific range of temperature and humidity values in which unexposed and exposed filters are to be conditioned for at least 24 hours immediately preceding their gravimetric analysis.

Confidence Interval — The numerical interval constructed around a point estimate of a population parameter, combined with a probability statement (the confidence coefficient) linking it to the population's true parameter value. If the same confidence interval construction technique and assumptions are used to

calculate future intervals, they will include the unknown population parameter with the same specified probability.

Confidentiality procedure — A procedure used to protect confidential business information (including proprietary data and personnel records) from unauthorized access.

Configuration — The functional, physical, and procedural characteristics of an item, experiment, or document.

Conformance — An affirmative indication or judgment that a product or service has met the requirements of the relevant specification, contract, or regulation; also, the state of meeting the requirements.

Consensus standard — A standard established by a group representing a cross section of a particular industry or trade, or a part thereof.

Contractor — Any organization or individual contracting to furnish services or items or to perform work.

Control chart - A graphical presentation of quality control (QC) information over a period of time. If a procedure is "in control," the results usually fall within established control limits. The chart is useful in detecting defective performance and abnormal trends or cycles, which can then be corrected promptly.

Corrective action - Any measures taken to rectify conditions adverse to quality and, where possible, to preclude their recurrence.

Correlation coefficient — A number between -1 and 1 that indicates the degree of linearity between two variables or sets of numbers. The closer to -1 or +1, the stronger the linear relationship between the two (i.e., the better the correlation). Values close to zero suggest no correlation between the two variables. The most common correlation coefficient is the product-moment, a measure of the degree of linear relationship between two variables.

Data Quality Objectives (DQOs) — The qualitative and quantitative statements derived from the DQO Process that clarify study's technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Data Quality Assessment (DQA) — The scientific and statistical evaluation of data to determine if data obtained from environmental operations are of the right type, quality, and quantity to support their intended use. The five steps of the DQA Process include: 1) reviewing the DQOs and sampling design, 2) conducting a preliminary data review, 3) selecting the statistical test, 4) verifying the assumptions of the statistical test, and 5) drawing conclusions from the data.

Data usability — The process of ensuring or determining whether the quality of the data produced meets the intended use of the data.

Data of known quality — Data that have the qualitative and quantitative components associated with their derivation documented appropriately for their intended use, and when such documentation is verifiable and defensible.

Data Quality Objectives (DQO) Process — A systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use. The key elements of the DQO process include:

Data reduction — The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collating them into a more useful form. Data reduction is irreversible and generally results in a reduced data set and an associated loss of detail.

Data Quality Indicators (DQIs) — The quantitative statistics and qualitative descriptors that are used to interpret the degree of acceptability or utility of data to the user. The principal data quality indicators are bias, precision, accuracy (bias is preferred), comparability, completeness, representativeness.

Deficiency — An unauthorized deviation from acceptable procedures or practices, or a defect in an item.

Demonstrated capability — The capability to meet a procurement's technical and quality specifications through evidence presented by the supplier to substantiate its claims and in a manner defined by the customer.

Design change — Any revision or alteration of the technical requirements defined by approved and issued design output documents and approved and issued changes thereto.

Design review — A documented evaluation by a team, including personnel such as the responsible designers, the client for whom the work or product is being designed, and a quality assurance (QA) representative but excluding the original designers, to determine if a proposed design will meet the established design criteria and perform as expected when implemented.

Design - The specifications, drawings, design criteria, and performance requirements. Also, the result of deliberate planning, analysis, mathematical manipulations, and design processes.

Detection Limit (DL) - A measure of the capability of an analytical method to distinguish samples that do not contain a specific analyte from samples that contain low concentrations of the analyte; the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability. DLs are analyte- and matrix-specific and may be laboratory-dependent.

Distribution — 1) The appointment of an environmental contaminant at a point over time, over an area, or within a volume; 2) a probability function (density function, mass function, or distribution function) used to describe a set of observations (statistical sample) or a population from which the observations are generated.

Document — Any written or pictorial information describing, defining, specifying, reporting, or certifying activities, requirements, procedures, or results.

Document control — The policies and procedures used by an organization to ensure that its documents and their revisions are proposed, reviewed, approved for release, inventoried, distributed, archived, stored, and retrieved in accordance with the organization's requirements.

Dry-bulb temperature -The actual temperature of the air, which is used for comparison with the wet-bulb temperature.

Duplicate samples — Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method, including sampling and analysis. See also *collocated sample*.

Electrostatic charge buildup- A buildup of static electrical charge on an item, such as the $PM_{2.5}$ filter, which makes it difficult to handle, attracts or repels particles, and can influence its proper weighing

Environmental technology — An all-inclusive term used to describe pollution control devices and systems, waste treatment processes and storage facilities, and site remediation technologies and their components that may be utilized to remove pollutants or contaminants from, or to prevent them from entering, the environment. Examples include wet scrubbers (air), soil washing (soil), granulated activated carbon unit (water), and filtration (air, water). Usually, this term applies to hardware-based systems; however, it can also apply to methods or techniques used for pollution prevention, pollutant reduction, or containment of contamination to prevent further movement of the contaminants, such as capping, solidification or vitrification, and biological treatment.

Environmental data — Any parameters or pieces of information collected or produced from measurements, analyses, or models of environmental processes, conditions, and effects of pollutants on human health and the ecology, including results from laboratory analyses or from experimental systems representing such processes and conditions.

Environmental programs — An all-inclusive term pertaining to any work or activities involving the environment, including but not limited to: characterization of environmental processes and conditions; environmental monitoring; environmental research and development; the design, construction, and operation of environmental technologies; and laboratory operations on environmental samples.

Environmental processes — Any manufactured or natural processes that produce discharges to, or that impact, the ambient environment.

Environmental monitoring — The process of measuring or collecting environmental data.

Environmental conditions — The description of a physical medium (e.g., air, water, soil, sediment) or a biological system expressed in terms of its physical, chemical, radiological, or biological characteristics.

Environmental data operations — Any work performed to obtain, use, or report information pertaining to environmental processes and conditions.

Equilibration chamber- A clean chamber usually constructed of plastic or glass, held at near constant temperature and humidity, used to store and condition $PM_{2.5}$ filters until they and their collected particulate sample (if the filters have been exposed) have reached a steady state of moisture equilibration.

Estimate — A characteristic from the sample from which inferences on parameters can be made.

Evidentiary records — Any records identified as part of litigation and subject to restricted access, custody, use, and disposal.

Expedited change — An abbreviated method of revising a document at the work location where the document is used when the normal change process would cause unnecessary or intolerable delay in the work.

Field blank filter- New filters, selected at random, that are weighed at the same time that presampling weights are determined for a set of $PM_{2.5}$ filters and used for QA purposes. These field blank filters are transported to the sampling site in the same manner as filter intended for sampling, installed in the sampler, removed from the sampler without sampling, stored in their protective containers inside the sampler's case at the sampling site until the corresponding exposed filter(s) is (are) retrieved, and returned for postsampling weighing in the laboratory, where it is handled in the same way as an actual sample filter and reweighed as a QC check to detect weight changes due to filter handling

Field blank — A blank used to provide information about contaminants that may be introduced during sample collection, storage, and transport. A clean sample, carried to the sampling site, exposed to sampling conditions, returned to the laboratory, and treated as an environmental sample.

Field (matrix) spike — A sample prepared at the sampling point (i.e., in the field) by adding a known mass of the target analyte to a specified amount of the sample. Field matrix spikes are used, for example, to determine the effect of the sample preservation, shipment, storage, and preparation on analyte recovery efficiency (the analytical bias).

Field split samples — Two or more representative portions taken from the same sample and submitted for analysis to different laboratories to estimate interlaboratory precision.

Field scientist- Refers to the ESAT contractor responsible for the PEP field activities

Financial assistance — The process by which funds are provided by one organization (usually governmental) to another organization for the purpose of performing work or furnishing services or items. Financial assistance mechanisms include grants, cooperative agreements, and governmental interagency agreements.

Finding — An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative, and is normally accompanied by specific examples of the observed condition.

Goodness-of-fit test — The application of the chi square distribution in comparing the frequency distribution of a statistic observed in a sample with the expected frequency distribution based on some theoretical model.

Grade — The category or rank given to entities having the same functional use but different requirements for quality.

Graded approach — The process of basing the level of application of managerial controls applied to an item or work according to the intended use of the results and the degree of confidence needed in the quality of the results. (See also *Data Quality Objectives (DQO) Process.*)

Guidance — A suggested practice that is not mandatory, intended as an aid or example in complying with a standard or requirement.

Guideline — A suggested practice that is not mandatory in programs intended to comply with a standard.

Hazardous waste — Any waste material that satisfies the definition of hazardous waste given in 40 CFR 261, "Identification and Listing of Hazardous Waste."

HEPA filter -A high efficiency particulate air filter is an extended-media dry-type filter with a minimum collection efficiency of 99.97% when tested with an aerosol of essentially monodisperse 0.3 µm particles.

Holding time — The period of time a sample may be stored prior to its required analysis. While exceeding the holding time does not necessarily negate the veracity of analytical results, it causes the qualifying or "flagging" of any data not meeting all of the specified acceptance criteria.

Hygrothermograph - Instrument resulting from the combination of a thermograph and a hygrograph and furnishing, on the same chart, simultaneous time recording of ambient temperature and humidity Laboratory blank filter New filters that are weighed at the time of determination of the presampling (tare) weight of each set of $PM_{2.5}$ filters intended for field use. These laboratory blank filters remain in the

laboratory in protective containers during the field sampling and are reweighed in each weighing session as a QC check.

Identification error — The misidentification of an analyte. In this error type, the contaminant of concern is unidentified and the measured concentration is incorrectly assigned to another contaminant.

Independent assessment — An assessment performed by a qualified individual, group, or organization that is not a part of the organization directly performing and accountable for the work being assessed.

Inspection — The examination or measurement of an item or activity to verify conformance to specific requirements.

Internal standard — A standard added to a test portion of a sample in a known amount and carried through the entire determination procedure as a reference for calibrating and controlling the precision and bias of the applied analytical method.

Item — An all-inclusive term used in place of the following: appurtenance, facility, sample, assembly, component, equipment, material, module, part, product, structure, subassembly, subsystem, system, unit, documented concepts, or data.

Laboratory analyst- The generic term used to describe the ESAT contractor(s) responsible for the actvities described in the standard operating procedures.

Laboratory split samples — Two or more representative portions taken from the same sample and analyzed by different laboratories to estimate the interlaboratory precision or variability and the data comparability.

Limit of quantitation — The minimum concentration of an analyte or category of analytes in a specific matrix that can be identified and quantified above the method detection limit and within specified limits of precision and bias during routine analytical operating conditions.

Management system — A structured, nontechnical system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for conducting work and producing items and services.

Management Systems Review (MSR) — The qualitative assessment of a data collection operation and/or organization(s) to establish whether the prevailing quality management structure, policies, practices, and procedures are adequate for ensuring that the type and quality of data needed are obtained.

Management — Those individuals directly responsible and accountable for planning, implementing, and assessing work.

Mass reference standard - NIST-traceable weighing standards, generally in the range of weights expected for the filters.

Matrix spike — A sample prepared by adding a known mass of a target analyte to a specified amount of matrix sample for which an independent estimate of the target analyte concentration is available. Spiked samples are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

May — When used in a sentence, a term denoting permission but not a necessity.

Mean squared error — A statistical term for variance added to the square of the bias.

Mean (arithmetic) — The sum of all the values of a set of measurements divided by the number of values in the set; a measure of central tendency.

Measurement and Testing Equipment (M&TE) — Tools, gauges, instruments, sampling devices, or systems used to calibrate, measure, test, or inspect in order to control or acquire data to verify conformance to specified requirements.

Memory effects error — The effect that a relatively high concentration sample has on the measurement of a lower concentration sample of the same analyte when the higher concentration sample precedes the lower concentration sample in the same analytical instrument.

Method — A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

Method blank — A blank prepared to represent the sample matrix as closely as possible and analyzed exactly like the calibration standards, samples, and quality control (QC) samples. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the analytical procedure.

Microbalance - A type of analytical balance that can weigh to the nearest 0.001 mg (that is, one microgram or one-millionth of a gram).

Mid-range check — A standard used to establish whether the middle of a measurement method's calibrated range is still within specifications.

Mixed waste — A hazardous waste material as defined by 40 CFR 261 Resource Conservation and Recovery Act (RCRA) and mixed with radioactive waste subject to the requirements of the Atomic Energy Act.

Must — When used in a sentence, a term denoting a requirement that has to be met.

Nonconformance — A deficiency in a characteristic, documentation, or procedure that renders the quality of an item or activity unacceptable or indeterminate; nonfulfillment of a specified requirement.

Objective evidence — Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measurements, or tests that can be verified.

Observation — An assessment conclusion that identifies a condition (either positive or negative) that does not represent a significant impact on an item or activity. An observation may identify a condition that has not yet caused a degradation of quality.

Organization structure — The responsibilities, authorities, and relationships, arranged in a pattern, through which an organization performs its functions.

Organization — A company, corporation, firm, enterprise, or institution, or part thereof, whether incorporated or not, public or private, that has its own functions and administration.

Outlier — An extreme observation that is shown to have a low probability of belonging to a specified data population.

Parameter — A quantity, usually unknown, such as a mean or a standard deviation characterizing a population. Commonly misused for "variable," "characteristic," or "property."

Peer review — A documented critical review of work generally beyond the state of the art or characterized by the existence of potential uncertainty. Conducted by qualified individuals (or an organization) who are independent of those who performed the work but collectively equivalent in technical expertise (i.e., peers) to those who performed the original work. Peer reviews are conducted to ensure that activities are technically adequate, competently performed, properly documented, and satisfy established technical and quality requirements. An in-depth assessment of the assumptions, calculations, extrapolations, alternate interpretations, methodology, acceptance criteria, and conclusions pertaining to specific work and of the documentation that supports them. Peer reviews provide an evaluation of a subject where quantitative methods of analysis or measures of success are unavailable or undefined, such as in research and development.

Performance Evaluation (PE) — A type of audit in which the quantitative data generated in a measurement system are obtained independently and compared with routinely obtained data to evaluate the proficiency of an analyst or laboratory.

$PM_{2.5}$ sampler - A sampler used for monitoring $PM_{2.5}$ in the atmosphere that collects a sample of particulate matter from the air based on principles of inertial separation and filtration. The sampler also maintains a constant sample flow rate and may record the actual flow rate and the total volume sampled. $PM_{2.5}$ mass concentration is calculated as the weight of the filter catch divided by the sampled volume. A sampler cannot calculate $PM_{2.5}$ concentration directly

$PM_{2.5}$- Particulate matter (suspended in the atmosphere) having an aerodynamic diameter less than or equal to a nominal 2.5 µm, as measured by a reference method based on 40 CFR Part 50, Appendix L, and designated in accordance with 40 CFR Part 53.

Pollution prevention — An organized, comprehensive effort to systematically reduce or eliminate pollutants or contaminants prior to their generation or their release or discharge into the environment.

Polonium-210 (^{210}Po) antistatic strip - A device containing a small amount of ^{210}Po that emits α particles (He^{2+}) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.

Polytetrafluoroethylene (PTFE)- The polymer that is used to manufacture the 46.2-mm diameter filters for $PM_{2.5}$ Federal Reference Method (FRM) and Federal Equivalent Method (FEM) samplers. Also known as Teflon®.

QA supervisor or coordinator - A staff member who assists in preparation of the reporting organization's quality plan, makes recommendations to management on quality issues (including training), oversees the quality system's control and audit components, and reports the results.

Population — The totality of items or units of material under consideration or study.

Precision — A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions expressed generally in terms of the standard deviation. Refer to *Appendix D, Data Quality Indicators,* for a more detailed definition.

Procedure — A specified way to perform an activity.

Process — A set of interrelated resources and activities that transforms inputs into outputs. Examples of processes include analysis, design, data collection, operation, fabrication, and calculation.

Project — An organized set of activities within a program.

Qualified services — An indication that suppliers providing services have been evaluated and determined to meet the technical and quality requirements of the client as provided by approved procurement documents and demonstrated by the supplier to the client's satisfaction.

Qualified data — Any data that have been modified or adjusted as part of statistical or mathematical evaluation, data validation, or data verification operations.

Quality control (QC) sample — An uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. Generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

Quality improvement — A management program for improving the quality of operations. Such management programs generally entail a formal mechanism for encouraging worker recommendations with timely management evaluation and feedback or implementation.

Quality management — That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality system.

Quality Control (QC) — The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality. The system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring the results are of acceptable quality.

Quality — The totality of features and characteristics of a product or service that bears on its ability to meet the stated or implied needs and expectations of the user.

Quality Assurance (QA) — An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Assurance Program Description/Plan — See *quality management plan*.

Quality Assurance Project Plan (QAPP) — A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria. The QAPP components are divided into four classes: 1) Project Management, 2) Measurement/Data Acquisition, 3) Assessment/Oversight, and 4) Data Validation and Usability. Guidance and requirements on preparation of QAPPs can be found in EPA QA/R-5 and QA/G-5.

Quality system — A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC).

Quality Management Plan (QMP) — A formal document that describes the quality system in terms of the organization's structure, the functional responsibilities of management and staff, the lines of authority, and the required interfaces for those planning, implementing, and assessing all activities conducted.

Radioactive waste — Waste material containing, or contaminated by, radionuclides, subject to the requirements of the Atomic Energy Act.

Readability - The smallest difference between two measured values that can be read on the microbalance display. The term "resolution" is a commonly used synonym.

Readiness review — A systematic, documented review of the readiness for the start-up or continued use of a facility, process, or activity. Readiness reviews are typically conducted before proceeding beyond project milestones and prior to initiation of a major phase of work.

Record (quality) — A document that furnishes objective evidence of the quality of items or activities and that has been verified and authenticated as technically complete and correct. Records may include photographs, drawings, magnetic tape, and other data recording media.

Recovery — The act of determining whether or not the methodology measures all of the analyte contained in a sample. Refer to *Appendix D, Data Quality Indicators*, for a more detailed definition.

Remediation — The process of reducing the concentration of a contaminant (or contaminants) in air, water, or soil media to a level that poses an acceptable risk to human health.

Repeatability - A measure of the ability of a microbalance to display the same result in repetitive weighings of the same mass under the same measurement conditions. The term “precision” is sometimes used as a synonym.

Repeatability — The degree of agreement between independent test results produced by the same analyst, using the same test method and equipment on random aliquots of the same sample within a short time period.

Reporting limit — The lowest concentration or amount of the target analyte required to be reported from a data collection project. Reporting limits are generally greater than detection limits and are usually not associated with a probability level.

Representativeness — A measure of the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. See also *Appendix D, Data Quality Indicators.*

Reproducibility — The precision, usually expressed as variance, that measures the variability among the results of measurements of the same sample at different laboratories.

Requirement — A formal statement of a need and the expected manner in which it is to be met.

Research (basic) — A process, the objective of which is to gain fuller knowledge or understanding of the fundamental aspects of phenomena and of observable facts without specific applications toward processes or products in mind.

Research (applied) — A process, the objective of which is to gain the knowledge or understanding necessary for determining the means by which a recognized and specific need may be met.

Research development/demonstration — The systematic use of the knowledge and understanding gained from research and directed toward the production of useful materials, devices, systems, or methods, including prototypes and processes.

Round-robin study — A method validation study involving a predetermined number of laboratories or analysts, all analyzing the same sample(s) by the same method. In a round-robin study, all results are compared and used to develop summary statistics such as interlaboratory precision and method bias or recovery efficiency.

Ruggedness study — The carefully ordered testing of an analytical method while making slight variations in test conditions (as might be expected in routine use) to determine how such variations affect test results. If a variation affects the results significantly, the method restrictions are tightened to minimize this variability.

Scientific method — The principles and processes regarded as necessary for scientific investigation, including rules for concept or hypothesis formulation, conduct of experiments, and validation of hypotheses by analysis of observations.

Self-assessment — The assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing and/or performing the work.

Sensitivity — the capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest. Refer to *Appendix D, Data Quality Indicators,* for a more detailed definition.

Service — The result generated by activities at the interface between the supplier and the customer, and the supplier internal activities to meet customer needs. Such activities in environmental programs include design, inspection, laboratory and/or field analysis, repair, and installation.

Shall — A term denoting a requirement that is mandatory whenever the criterion for conformance with the specification permits no deviation. This term does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

Should — A term denoting a guideline or recommendation whenever noncompliance with the specification is permissible.

Significant condition — Any state, status, incident, or situation of an environmental process or condition, or environmental technology in which the work being performed will be adversely affected sufficiently to require corrective action to satisfy quality objectives or specifications and safety requirements.

Software life cycle — The period of time that starts when a software product is conceived and ends when the software product is no longer available for routine use. The software life cycle typically includes a requirement phase, a design phase, an implementation phase, a test phase, an installation and check-out phase, an operation and maintenance phase, and sometimes a retirement phase.

Source reduction — Any practice that reduces the quantity of hazardous substances, contaminants, or pollutants.

Span check — A standard used to establish that a measurement method is not deviating from its calibrated range.

Specification — A document stating requirements and referring to or including drawings or other relevant documents. Specifications should indicate the means and criteria for determining conformance.

Spike — A substance that is added to an environmental sample to increase the concentration of target analytes by known amounts; used to assess measurement accuracy (spike recovery). Spike duplicates are used to assess measurement precision.

Split samples — Two or more representative portions taken from one sample in the field or in the laboratory and analyzed by different analysts or laboratories. Split samples are quality control (QC) samples that are used to assess analytical variability and comparability.

Standard Operating Procedure (SOP) — A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps and that is officially approved as the method for performing certain routine or repetitive tasks.

Standard deviation — A measure of the dispersion or imprecision of a sample or population distribution expressed as the positive square root of the variance and has the same unit of measurement as the mean.

Supplier — Any individual or organization furnishing items or services or performing work according to a procurement document or a financial assistance agreement. An all-inclusive term used in place of any of the following: vendor, seller, contractor, subcontractor, fabricator, or consultant.

Surrogate spike or analyte — A pure substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them to establish that the analytical method has been performed properly.

Surveillance (quality) — Continual or frequent monitoring and verification of the status of an entity and the analysis of records to ensure that specified requirements are being fulfilled.

Technical Systems Audit (TSA) — A thorough, systematic, on-site qualitative audit of facilities, equipment, personnel, training, procedures, recordkeeping, data validation, data management, and reporting aspects of a system.

Technical review — A documented critical review of work that has been performed within the state of the art. The review is accomplished by one or more qualified reviewers who are independent of those who performed the work but are collectively equivalent in technical expertise to those who performed the original work. The review is an in-depth analysis and evaluation of documents, activities, material, data, or items that require technical verification or validation for applicability, correctness, adequacy, completeness, and assurance that established requirements have been satisfied.

Traceability — The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Traceability - The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, all having stated uncertainties. Many quality assurance programs demand traceability of standards to a national standard. In most cases this can be achieved through a standard traceable to NIST.

Trip blank — A clean sample of a matrix that is taken to the sampling site and transported to the laboratory for analysis without having been exposed to sampling procedures.

Validation — Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use have been fulfilled. In design and development, validation concerns the process of examining a product or result to determine conformance to user needs. See also *Appendix G, Data Management.*

Variance (statistical) — A measure or dispersion of a sample or population distribution. Population variance is the sum of squares of deviation from the mean divided by the population size (number of elements). Sample variance is the sum of squares of deviations from the mean divided by the degrees of freedom (number of observations minus one).

Verification — Confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, verification concerns the process of examining a result of a given activity to determine conformance to the stated requirements for that activity.

Wet-bulb thermometer - A thermometer with a muslin-covered bulb, which is moistened and which is used to measure the wet-bulb temperature.

Wet-bulb temperature - The temperature of the wet-bulb thermometer at equilibrium with a constant flow of ambient air at a rate of from 2.5 to 10.0 meters per second.

Appendix B
Data Qualifiers/ Flags

A sample qualifier or a result qualifier consists of 3 alphanumeric characters which act as an indicator of the fact and the reason that the subject analysis (a) did not produce a numeric result, (b) produced a numeric result but it is qualified in some respect relating to the type or validity of the result or produced a numeric result but for administrative reasons is not to be reported outside the laboratory.

Field Qualifiers

Code	Definition	Description
CON	Contamination	Contamination incuding observations of insects or other debris
DAM	Filter Damage	Filter appeared damaged
EST 1/	Elapsed Sample Time	Elapsed sample time out of specification
EVT	Event	exceptional event expected to have effected sample (dust, fire , spraying etc)
FAC	field accident	There was an accident in the field that either destroyed the sample or rendered it not suitable for analysis.
FAT	Failed Temperature Check Ambient	Ambient temperature check out of specification
FIT	Failed Temperature Check Internal	Internal temperature check out of specification
FLR 1/	Flow Rate	Flow rate 5 min avg out of specification
FLT 1/	Filter Temperature	Filter temperature differential, 30 second interval out of specification
FMC	Failed Multi point Calibration Verification	Failed the initial Multi point calibration verification
FPC	Failed Pressure Check	Barometric pressure check out of specification
FSC	Failed Single Point Calibration Verification	Failed the initial single point calibration verification
FVL	Flow volume	Flow volume suspect
LEK	Leak suspected	internal/external leak suspected
SDM	Sampler Damaged	Sampler appears to be damaged which may have effected filter

1/- Flag generated by sampling equipment

Laboratory Qualifiers

Code	Definition	Explanation
ALT	alternate measurement	The subject parameter was determined using an alternate measurement method. Value is believed to be accurate but could be suspect.
AVG	average value	Average value - used to report a range of values
BDL	below detectable limits	There was not a sufficient concentration of the parameter in the sample to exceed the lower detection limit in force at the time the analysis was performed. Numeric results field, if present is at best, an approximate value.
BLQ	below limit of quantitation	The sample was considered above the detection limit but there was not a sufficient concentration of the parameter in the sample to exceed the lower quantitation limit in force at the time the analysis was performed.
CAN	canceled	The analysis of this parameter was canceled and not preformed.
CBC	cannot be calculated	The calculated analysis result cannot be calculated because an operand value is qualified.

Code	Definition	Explanation
EER	entry error	The recorded value is known to be incorrect but the correct value cannot be determined to enter a correction.
FBK	found in blank	The subject parameter had a measurable value above the established QC limit when a blank was analyzed using the same equipment and analytical method. Therefore, the reported value may be erroneous.
FCS	failed collocated sample	Collocated sample exceeded acceptance criteria limits.
FFB	failed field blank	Field blank samples exceeded acceptance criteria limits.
FIS	failed internal standard	Internal standards exceeded acceptance criteria limits.
FLB	failed laboratory blank	Laboratory blank samples exceeded acceptance criteria limits.
FLD	failed laboratory duplicate	Laboratory duplicate samples exceeded acceptance criteria limits.
FLH	failed laboratory humidity	Laboratory humidity exceeded acceptance criteria limits
FLT	failed laboratory temperature	Laboratory temperature exceeded acceptance criteria limits.
FQC	failed quality control	The analysis result is not reliable because quality control criteria were exceeded when the analysis was conducted. Numeric field, if present, is estimated value.
FRW	failed replicate weight	The sample was reweighed and was not repeatable with acceptance criteria.
HTE	holding time exceeded	Filter holding time exceeded acceptance criteria limits
ISP	improper sample preservation	Due to improper preservation of the sample, it was rendered not suitable for analysis.
LAC	laboratory accident	There was an accident in the laboratory that either destroyed the sample or rendered it not suitable for analysis.
LLS	less than lower standard	The analysis value is less than the lower quality control standard.
LTC	less than criteria of detection	Value reported is less than the criteria of detection (which may differ from instrument detection limits).
NAR	no analysis result	There is no analysis result required for this subject parameter. (no result; no storet remark)
PSD	possible shipping damage	Upon receipt of filter from the field, the filter appears to be damaged during shipping.
REJ	rejected	The analysis results have been rejected for an unspecified reason by the laboratory. For any results where a mean is being determined, this data was not utilized in the calculation of the mean.
REQ	reque for re-analysis	The analysis is not approved and must be re-analyzed using a different method.
RET	return(ed) for re-analysis	The analysis result is not approved by laboratory management and reanalysis is required by the bench analyst with no change in the method.
RIN	re-analyzed	The indicated analysis results were generated from a re-analysis
SIS	sample integrity suspect	Based upon other flags or free form notesthe data quilty from this sample is suspect.

Code	Definition	Explanation
STD	internal standard	The subject parameter is being utilized as an internal standard for other subject parameters in the sample. There is no analysis result report, although the theoretical and/or limit value(s) may be present.
UND	analyzed but undetected	Indicates material was analyzed for but not detected.
VOD	Void sample	The sample had flags indicating that the sample integrity was suspect and after examination, conluded to halt further processing